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of the
SOCIETY OF DYERS AND COLOURISTS

Volume 67 Number 3

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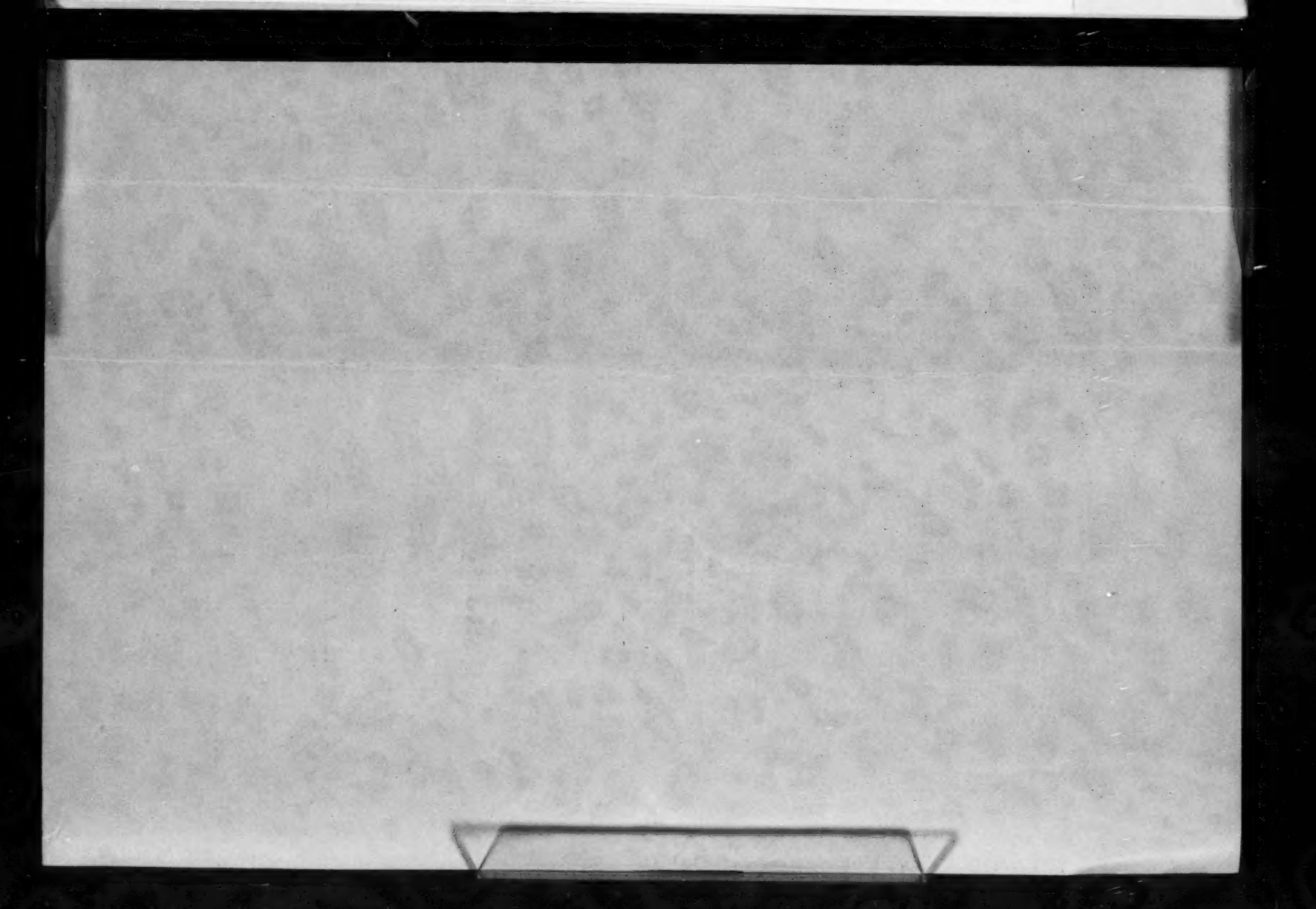

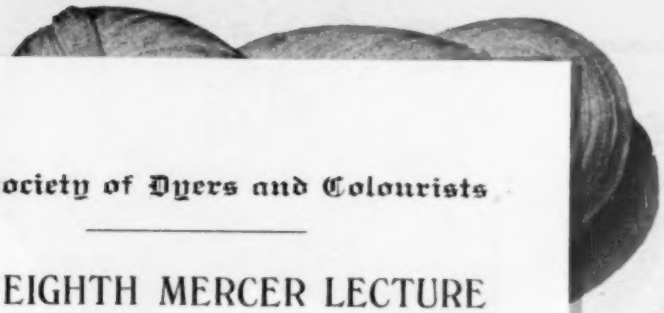

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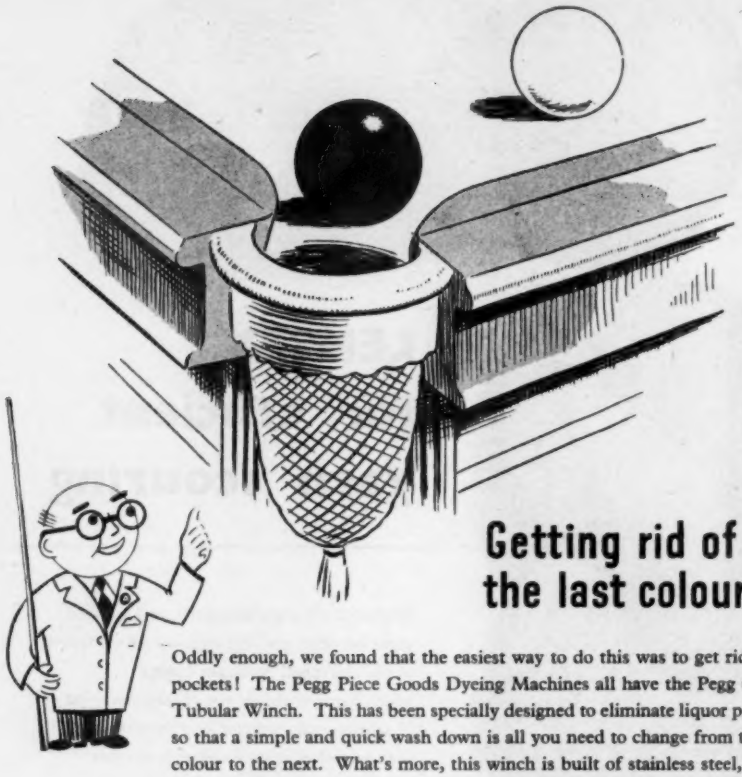

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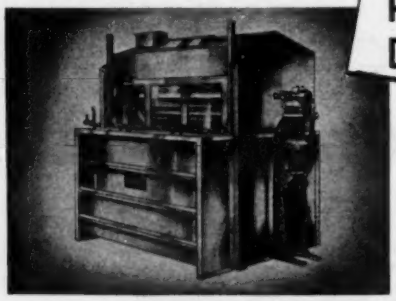

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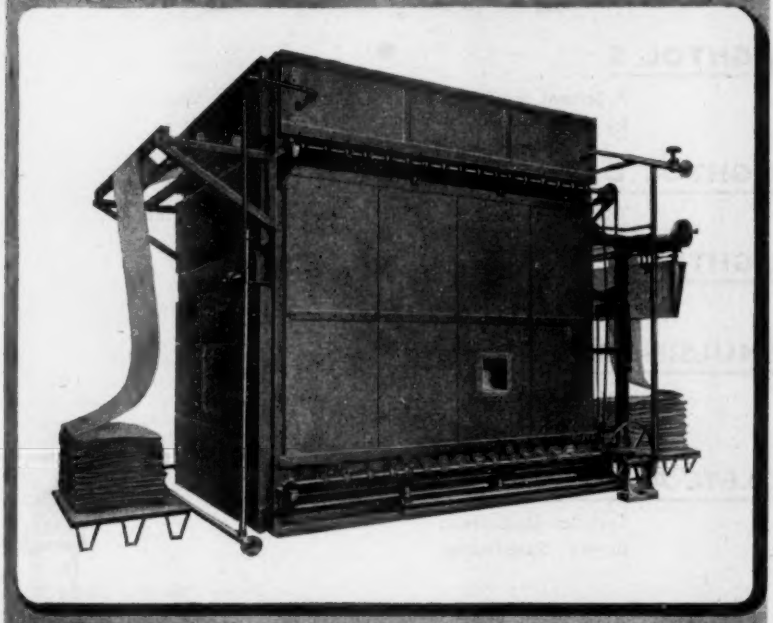

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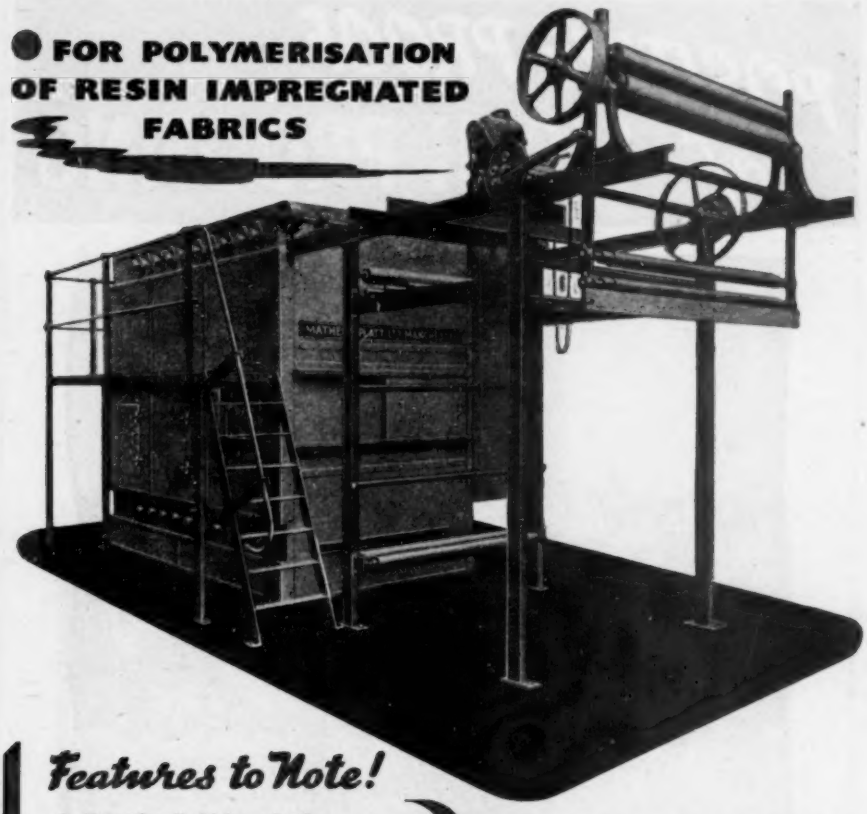

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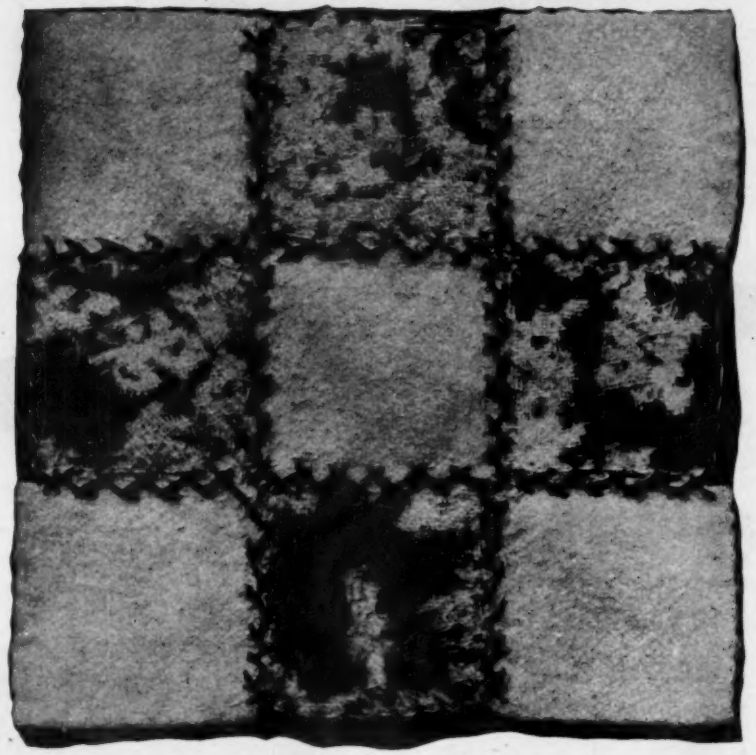

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1951 and pages 345–348 of the July 1950 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 32–34 Piccadilly, Bradford, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURE

Fast Shades on Nylon Textiles — *G. T. Douglas*

COMMUNICATIONS

Some Observations on the Hardening Process used in Felt Manufacture. I— Factors influencing Hardening
R. S. M. Frohnsdorff and C. S. Whewell

Comparative Tests in Shrink-resisting Men's Half Hose
J. R. Stewart

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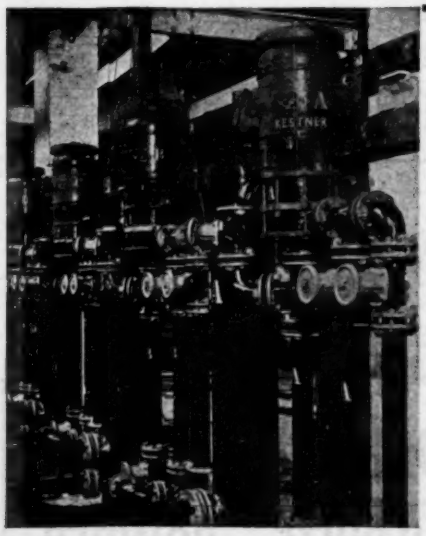

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Forthcoming Meetings of the Society

1951

Friday 30th Mar. — ANNUAL GENERAL MEETING AND DINNER OF THE SOCIETY, GRAND HOTEL LEICESTER

Friday 20th April — EIGHTH MERCER LECTURE—Dr. D. Traill, St. Enoch Hotel, Glasgow

HUDDERSFIELD SECTION

All meetings held at Field's Cafe, Westgate, Huddersfield, at 7.30 p.m. unless otherwise stated

1951

Tuesday 20th Mar. — F. Jackson, Esq. *Water Treatment for Boilers and Processing*

Tuesday 10th April — ANNUAL GENERAL MEETING Miss Esmee Smith. *Union Dyeing with Special Reference to Modern Fibres*

MIDLANDS SECTION

All meetings commence at 7 p.m. (except where stated otherwise)

1951

Wednesday 21st Mar. 7.15 p.m. — *Recent developments in fluorescent lamps, with particular regard to colour problems.* Dr. S. T. Henderson (Joint meeting with British Association of Chemists). School of Arts and Crafts, Derby

Wednesday 18th April — ANNUAL GENERAL MEETING. Followed by D. Carter, M.Sc., F.R.I.C. *Some impressions of American and Continental Dyehouses* King's Head Hotel, Loughborough

SCOTTISH SECTION

All meetings at 7 p.m. in St. Enoch's Hotel, Glasgow, unless otherwise stated

1951

Tuesday 20th March — ANNUAL GENERAL MEETING. Followed by ordinary meeting at 7.30 p.m. Details to be announced later.

BRADFORD JUNIOR BRANCH

All meetings to be held in the Technical College, Bradford, at 7.15 p.m.

1951

Tuesday 20th March — STUDENTS' NIGHT

Monday 16th April — ANNUAL GENERAL MEETING

MANCHESTER JUNIOR BRANCH

All meetings held in the Reynold's Hall, College of Technology, Manchester, at 6.30 p.m.

1951

Tuesday 3rd April — S. R. Cockett, M.Sc.(Tech.), F.R.I.C., of the Bradford Dyers' Association. *Detection and Correction of Faults in Dyed Materials*

NORTHERN IRELAND SECTION

All meetings to be held in Queen's Hotel, Belfast, at 7.30 p.m.

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March — Annual General Meeting and Dinner (Date to be announced later)

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THE JOURNAL
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Volume 67—Number 3 MARCH 1951 *Issued Monthly*

COMMUNICATIONS

Some Aspects of the Drying and Heating of Textiles

IV—The Effect of Drying Conditions on the Latent Strain in the Yarn of Viscose Cakes

J. M. PRESTON and J. C. CHEN

The effects of drying viscose rayon yarn in cake form by convection, infra-red, and high-frequency methods have been compared.

INTRODUCTION

In the pristine state regenerated cellulose fibres are highly swollen. Even after washing and centrifuging the water content of a viscose cake is 50–60% by weight of the moist material. By volume the water content is even higher, about 75%. As most of this water is in the fibres and occupying its usual volume[1], it follows that considerable shrinkage must take place on drying. Cellulose fibres are markedly anisotropic, and only a small fraction of the swelling occurs axially. Were it not for this, the drying of a textile material in an effectively strain-free condition would be very difficult indeed, as the greater the axial shrinkage the more difficult it becomes to support it during drying without introducing strain. The axial shrinkage of the more common viscose fibres is 1–7%, the exact figure depending mainly upon the degree of orientation. The appreciable axial shrinkage on drying causes serious problems. When the yarn is wound on a bobbin the core prevents the shrinkage of the innermost layers on drying, because the core prevents the natural contraction and causes a latent strain in proportion to the contraction that would have occurred if the yarn had been free from restraint. This latent strain is released on wetting the yarn.

With cakes there is no solid core and the innermost layers can contract freely, but, as with bobbins also, the outermost layers are liable to be dried under strain. This happens when the outer layers are dried whilst the inner layers are still wet and turgid. In this state the inner layers act like a solid core. Of course, this is avoided if the inner layers are dried first, so that they become flaccid and can be easily compressed as the outer layers contract. The prime factor in obtaining yarn free from latent strain is that it shall be dried under conditions that permit free contraction. In effect this demands two main conditions in drying a yarn package. Firstly the inner layer of yarn must not be restrained by any core. Secondly the drying must proceed from inside outwards. In addition, there is a secondary condition in obtaining yarn free from tension. The first time yarn is dried it does not attain its final stable length, but tends to contract a little further each time it is wetted and re-dried[2,3]. The structure of regenerated cellulose appears to consolidate progressively with repeated drying, wetting, and re-drying, as shown by the progressively diminishing swelling in water[4]. Steam accelerates this consolidation process[5].

Experiments were carried out on the drying of freshly spun viscose cakes to test the best conditions for obtaining yarn free from latent strain.

EXPERIMENTAL

Fresh cakes washed free from acid were dried in a variety of ways; subsequently the cakes were unwound, and samples of yarn were taken at successive layers of the cake and tested. Latent strain was measured by hanging lengths of yarn in an apparatus which could be used to wet the lengths of yarn and to dry them again subsequently[6]. The lengths of yarns (approx. 1 m. each) were measured accurately dry; they were then wetted thoroughly, and the lengths remeasured; finally the yarns were dried at 60°C., and the lengths measured again in this state. The difference between the first (dry) and last (redried) lengths gives the latent strain which is released by wetting and redrying, whilst the difference between the wet and redried lengths gives the reversible swelling.

The data for the latent strains of twelve cakes dried in different ways are plotted in Fig. 1–3. The first two figures contain data on cakes dried by the conventional methods of radiation and convection. Fig. 3 contains data on cakes dried in a high-frequency (11 megacycles/sec.) electric field. The

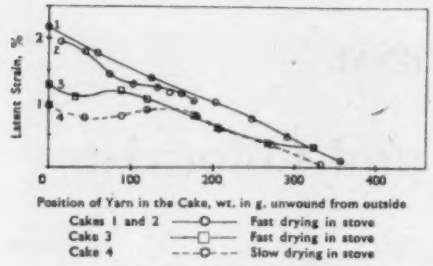
Latent Strain, %
Position of Yarn in the Cake, wt. in g. unwound from outside
Cakes 1 and 2 — Fast drying in stove
Cake 3 — Fast drying in stove
Cake 4 — Slow drying in stove

FIG. 1—Viscose Cakes dried commercially

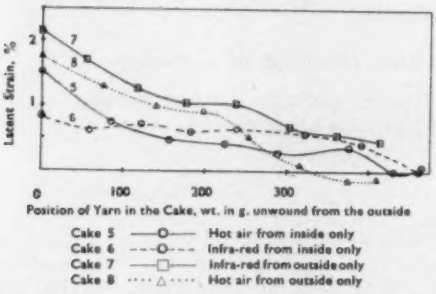
Latent Strain, %
Position of Yarn in the Cake, wt. in g. unwound from the outside
Cake 5 — Hot air from inside only
Cake 6 — Infra-red from inside only
Cake 7 — Infra-red from outside only
Cake 8 — Hot air from outside only

FIG. 2—Viscose Cakes dried experimentally by Convection and Radiation

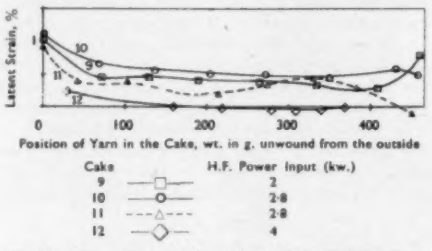

FIG. 3—Viscose Cakes dried experimentally by High Frequency

general features of Fig. 1 and 2 indicate a progressive diminution of strain from the outside to the inside of the cakes. With the cake dried with infra-red from the inside there is a much smaller gradient than with the other cakes. The data of Fig. 3 show that the cakes dried by a high-frequency electric field exhibit small differences of latent strain from outside to inside and generally low values of the strain.

Cakes 1–3 were dried commercially in 2–3 days, and cake 4 was dried commercially in about 10 days.

All the remaining cakes were dried under various experimental conditions in the laboratory. Cake 5 was dried by hot air in a stove; the outside and ends of the cake were protected by impermeable covers, so that drying could occur only from the inner surface of the cake. The latent strain is seen to be less uneven than with cake 8, which was dried from the outside only. In both cases the hot air reaching the cake was at about 60°C. Cake 6 was dried by "infra-red" radiation provided by a 70-watt carbon-filament bulb placed in the centre of the cake, which was arranged with its axis vertical. The drying temperatures in the cake were observed by means of three 40 s.w.g. copper–constantan couples placed in the inside, the centre, and the outside of the wall of the cake. The outer temperature rose to about 33°C., fell below 30°C., and rose again to over 35°C. during the drying. The central couple showed similar temperature changes at a higher level, viz. 37°, 32°, and 60°C. The innermost couple was the hottest and rose progressively from 38° to 88°C. during the drying. This cake has the least variation of latent strain of any of the cakes dried by convection and radiation. Cake 7 was dried from the outside with four infra-red lamps consuming a total of about 700 w. Temperatures were again recorded at three positions in the cake. On the average they were much higher, corresponding to the greater power input. In this experiment the temperatures were highest on the outside, but only exceeded 90°C. slightly in the outermost layer when the moisture content of the cake had been reduced to about 10%.

Cakes 9–12 were dried by high-frequency (H.F.) heating. The frequency was 11 megacycles, power inputs of 2–4·5 kw. being used. In operation the cake was supported on an electrode made of wire gauze stretched across a circular metal frame slightly larger in diameter than the cakes. The electrode was held up by means of nylon cords attached to a spring balance. The upper electrode was of similar construction to the lower one. It rested lightly on the top of the cake. Metal gauze electrodes were used to permit the freest possible escape of water vapour and to reduce the weight of metal that had to be heated by the escaping vapour with a consequent reduction in condensation. Curves for the latent strain in these cakes are given in Fig. 3. It will be seen that the optimum results are obtained with the highest power input used. During the drying the power input and weight of the cakes were observed at intervals without interrupting the drying. Observations of the temperature were attempted with both a thermocouple and a thermometer, but the former had to be abandoned as under the conditions used it set fire to the cake. The thermometer caused no trouble and showed no significant heating when placed in the H.F. field by itself.

The temperature–water content curve for a cake dried with a mean H.F. power input of 1·9 kw. is given in Fig. 4. It will be seen that the power input was so high that the temperature rose rapidly to the boiling point, little water being removed during this initial heating-up stage. Subsequently the moisture content fell rapidly, and the temperature rose steadily till it reached 130°C. at a stage when the cake was almost completely dry. The conditions of drying here were different from those with any of the other methods, since with H.F. the power input can be made so high that the water is rapidly vaporised, and not evaporated at temperatures below the normal boiling point and therefore at below atmospheric pressure.

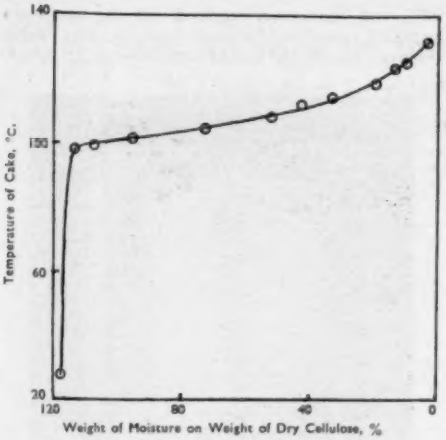

FIG. 4

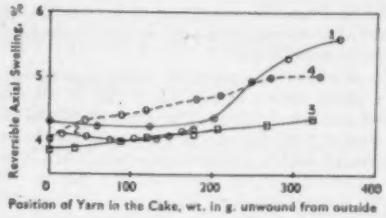

FIG. 6

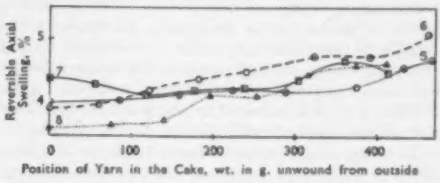

FIG. 7

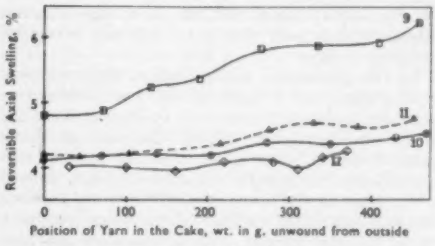

FIG. 8

As it appeared that the rise of temperature above 100°C. in the H.F. drying might be associated with the difficulty of escape of the steam, an experiment was performed with a block of cellulose gel, which had been prepared by the spontaneous coagulation of viscose. A piece about 5 cm. × 5 cm. × 2 cm. had been washed with hot water at intervals over a period of several months to remove soluble salts. Superfluous moisture was mopped off, and the block was drilled with a small hole to house a thermometer. It was then dried by H.F. heating. The temperature rose rapidly and finally reached 180°C., although the block was still not quite dry.

Cakes were dried at various H.F. power inputs. The product of mean power input and drying time, reduced to a basis of equal amounts of water evaporated for eight cakes, was found to have a mean value of 1·38 kWh. of H.F. input per kilogram of water dried out of the cakes. This is equivalent to very nearly 50% efficiency and is slightly better than the figure quoted by Fleming[7]. The relation of power input to drying time should be hyperbolic, so that a plot of power against reciprocal time should lie on a straight line. The data for eight cakes are plotted in this way in Fig. 5, the times referring to cakes each containing 0·50 kg. of water.

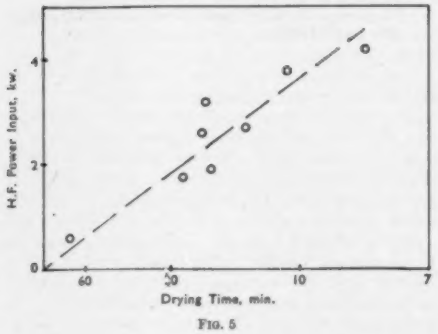

FIG. 5

It will be seen that there is a considerable scatter of points, but that they lie about a straight line passing through the origin.

The reversible swelling data of various cakes are given in Fig. 6–8. The values range about a rough mean of 4–4·5%. In all cases there is a more or less marked increase from the outside to the inside of the cake. Such a trend is to be expected, since the tension decreases during spinning as the internal diameter of the cake diminishes, unless some method of compensation is employed. It is interesting to note that the cakes (10–12) dried at the highest power inputs have the most uniform swelling values throughout.

DISCUSSION

In the drying of the cakes by convection or radiation the water is evaporated at temperatures well below the boiling point because the energy input to the moist fibres is low, but in H.F. drying the energy input can be made so high that the water can be vaporised rapidly. In the H.F. drying experiments with the cakes the water was driven off in a few minutes, in marked contrast with the days required in normal drying methods, and the cakes were surrounded with a cloud of steam escaping in all directions. Under these conditions the generation of water vapour is disseminated throughout the cake, which is entirely

different from the conditions of low energy input, where slow evaporation occurs only at the surfaces of the exposed fibres.

In H.F. drying steam is being generated not only in all layers of the cakes but at all depths inside individual fibres. The attempts of the steam to escape, and thereby to remove from the cakes the energy which has been absorbed, is hindered by the structures of the fibres and the yarns. These create a back pressure, which may be expected to increase as the structures of the fibres shrink during drying. It is therefore to be expected that the temperature will rise above the normal boiling point of water, as is observed. Increase of the length of the path that must be followed by the escaping steam should increase the back pressure and the temperature on this hypothesis. Confirmation of this is found in the great temperature rise of the block of cellulose gel during drying.

Heating moist viscose fibres to high temperatures is known to facilitate the release of latent strain and promote uniformity[5,8]. It is probably for this reason that cakes 10–12, dried at the highest energy inputs used, exhibit both the smallest latent strains and the most uniform reversible swelling values.

In the convection and radiation drying experiments, Fig. 1 and 2 show that the best results were obtained by slow drying and by drying from the inside preferentially; indeed the best of these, cakes 4 and 5, were as good as the poorest H.F. cake, No. 9. Even with cakes dried by H.F. there is a tendency for the latent strain to increase towards the exposed outer surface of the cake, probably owing to the greater vapour pressure gradient between the fibres and their environments in these regions. This will promote more rapid drying from these layers, although the energy input was originally uniform throughout the cake, since there is a temperature gradient falling towards the exposed layers of the cake[9].

It was observed in unwinding the yarns from the various cakes that there was buckling of the inside layers with the formation of channels as seen in Fig. 9. This indicates that the outside layers had begun to contract before the inside layers. It is not clear how this could have occurred with cakes dried from the inside or by H.F.

The data for the cakes dried by H.F. show that it is possible to dry cakes substantially free from strain. The efficiency of conversion of H.F. energy to water evaporated was found to range from 55 to 82%, but the power required for the necessary auxiliary circuits, filament heating, and valve cooling reduced the overall efficiency to 22–33%*.

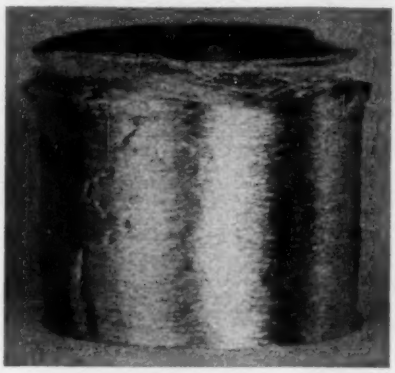

FIG. 9

Under present conditions H.F. drying is probably too expensive for general purposes. The next best methods of drying cakes judged by the standard of freedom from latent strain are drying from the inside and very slow drying.

* Some much higher overall efficiencies have been claimed elsewhere[10].

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Some Aspects of the Drying and Heating of Textiles

V—Migration in relation to Moisture Content

J. M. PRESTON and A. BENNETT

Migration which occurs during drying has been studied for different textile materials and different moisture contents. The greater the moisture content the more marked is the migration, and there is a characteristic lower limit at which it ceases.

INTRODUCTION

An earlier paper[1] described the observation that during drying there was a sudden rise of temperature when the surface moisture had evaporated. This occurred under conditions of rapid drying by radiant heating and can be accounted for by a change in the area of water covering fibres from the whole area of the latter to that fraction of the fibres not occupied by fibre substance. Before this stage in the drying there is a continuous film of water covering the fibres; i.e. there is excess of water above that required to saturate the fibres. Under these conditions fibre-to-fibre contacts provide a continuous film of water throughout an assembly of fibres. This permits liquid migration effects to take place[2]. It appeared probable that evidence of migration could be used conveniently as evidence of excess water above the saturation level. The water content at which migration ceases should then indicate the "critical moisture content"[1]. On the practical side this information should be useful in attempting to control or eliminate migration effects.

EXPERIMENTAL

Using the technique described previously in studying migration phenomena, suitable non-substantive coloured indicators were required. Azogeranine 2GS (*C.I.* 31) was selected for glass and cellulosic fibres; similarly Chlorazol Black BH (*C.I.* 401) or Chlorazol Black E (*C.I.* 581) with the addition of a sulphated fatty alcohol was used with wool and nylon materials.

Since rapid drying had previously been shown to produce striking migration effects[2], the first series of experiments were made under conditions of rapid drying. Trials were carried out using "infra-red" heating and drying in a hot-air stove. Both showed similar migration effects to those previously reported[2]. In addition new information was obtained about the effect of water content. It was found, as had been expected, that fabrics with the highest water content showed the greatest migration. Such a series run on cotton and dried by "infra-red" from one side only is shown in Fig. 1. To show the migration effect the corners have been turned over and pinned down. The water contents range from 34 to 70% on the weight of the dry cotton. It is seen that the migration effect diminishes with water content. (The lighter overall shades of the samples which had lower water contents and, correspondingly, lower dye contents, are due to using a uniform concentration of Azogeranine solution for all the samples.) Various series of drying experiments were carried out, with similar results to those just described. Different fabrics were used, and the interesting observation was made that open raised fabrics showed the effect of migration less strikingly than compact closely woven ones.

It was found that rapid drying conditions presented serious difficulties when it was desired to start drying with precisely known water contents. This was because appreciable drying occurs whilst the samples are being arranged. Furthermore, ill-defined transient effects take place during the initial stages as the samples are heating up when first entered into the stove or placed before the "infra-red" radiator. By elaborating the technique these difficulties might have been overcome, but there remained the migration effect through the fabric caused by the temperature gradient, which raises the water content of the cooler side of the fabric, as will be shown later. For these reasons it was decided to study the effects of drying at room temperature. It was found that drying at room temperature under proper conditions produced clear migration, and had several advantages— Drying was slow and the change of water content during the arrangement of the samples was estimated to be not more than 1% with light fabrics and less with heavier ones. A second advantage, with one-sided drying, was that the temperature gradient through the fabric was small, being not more than that equivalent to the wet-bulb thermometer effect and falling towards the surface of evaporation. Because of this the small vapour pressure gradient could transfer only a small amount of water, equivalent to that evaporated, and, as the transfer is toward the drying surface, the water content could not rise above the initial value in any part of the fabric.

The samples were prepared by soaking weighed, conveniently sized pieces of oven-dry fabric in a solution of non-substantive colouring matter, centrifuging for a short time, and then taking individual samples, hanging them up in the air, and allowing to dry slowly. After a period of time judged appropriate, the samples were placed in weighing bottles and weighed. When the weight indicated the desired water content, the samples were left in the stoppered weighing bottles and allowed to stand at a steady temperature for several days so that diffusion would make the samples as uniform as possible. After rechecking the weight, each sample of fabric was now taken in turn from its bottle and laid on a glass plate; a small circle of glass 3·8 cm. in diameter was placed on the fabric with an additional weight on top making a load of 50 g. in all. The assemblies were placed in a cupboard, to avoid draughts and stray radiation effects, and allowed to dry slowly at room temperature.

Drying takes place at the uncovered surface of the fabric. If there is any migration of liquid water, then the indicating dye will be carried with it, and the part under the glass will be partly stripped of colour. Some of the results of experiments on glass, viscose, cotton, and wool fabrics are shown in Fig. 2–6. From the results for glass shown in Fig. 2, it will be seen that it is not until the water content is reduced to about 3% of the weight of the dry material that migration is almost completely stopped. There is still evidence of very slight migration, but compared with the sample which had 7% water content, the effect is negligible. Similarly the viscose samples photographed in Fig. 3 show that migration had almost ceased with a starting water content of 42%. This is confirmed by the experiment photographed in Fig. 4, in which it will be seen that there had been slight migration at 41% but none at 39%. The results of similar experiments with cotton and wool fabrics are shown in Fig. 5 and 6 respectively. It will be noticed that with cotton very marked migration occurs at a water content lower than that at which all migration had ceased in the case of wool or viscose rayon.

The results of these and other series of experiments are summarised in the accompanying table.

Fibre	Water Content (% on wt. of dry material) Cessation of Migration	Critical Moisture Content[1]	Break Point in H.F. Drying[2]
Viscose	40 ± 1	38	38, 40
Wool	36 ± 3	39	35
Cotton	21 ± 2	26	28
Nylon	10 ± 1	—	7
Glass	< 3	6	2

The second column gives the water contents at which migration ceases. The third and fourth columns present comparable data from the literature on drying, in which different criteria have been taken to indicate the removal of surface water. The three columns of data are in reasonable agreement.

Earlier it was noted that heating one side of a partly covered fabric showed the most marked migration effects on the unheated side. This was thought to be because the water content of this side was temporarily raised, owing to the diffusion of water vapour from the heated side to the unheated side, where it was condensed on the fibres. The effect was greatest if the cool side of the fabric rested on a cool, impermeable support. With this in mind it was supposed that, even if the water content was originally below that required for migration, the content of the cool side could be temporarily raised sufficiently for migration to take place on that side of the fabric. Experiments were designed to test this supposition. Pieces of heavy cotton collar fabric were soaked in Azogeranine 2G solution, centrifuged to remove as much liquid as possible, and then allowed to dry freely in the laboratory. Each was then conditioned in an atmosphere of 64% R.H. for a week, after which the water content was about 8%. This is greatly below the level, 21%, required for normal migration effects.

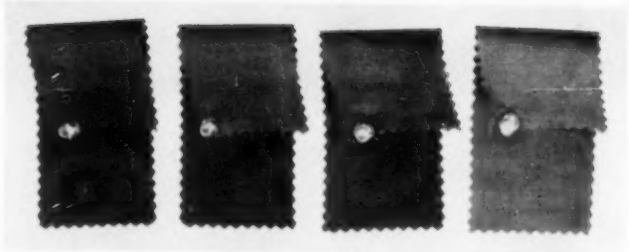
The conditioned fabric was laid on a glass plate; a brass cylinder 5·3 cm. in diameter and 5·1 cm. high was heated, and at about 90°c. was placed on the fabric. After cooling to room temperature the cylinder was removed and the fabric examined. No signs of migration were seen on the face in contact with the cylinder, as will be seen from the photograph reproduced in Fig. 7. The reverse side showed migration. There was a lighter circle corresponding in size to the diameter of the brass cylinder, as will be seen from Fig. 8. This result was expected on the basis of the mechanism propounded above, but varying the conditions slightly produced a surprising result. In the modified experiment a brass cylinder was used as before, but in this case it had an axial hole 0·6 cm. in diameter and 0·7 cm. deep at the centre in the bottom of the cylinder. When this was placed on the cotton fabric as before the appearances of the face and back of the fabric were as shown in Fig. 9 and 10. As can be seen from the former, where the fabric was in contact with the cylinder there was no migration on the face. In the latter figure it will be seen that there had been migration in the corresponding position on the back of the fabric as in Fig. 8. The surprising result occurred at the position of the fabric opposite the hole in the cylinder. Very marked migration had occurred away from the centre on both the front and the back of the fabric. Corresponding in position to the rim of the central hole there is a ring of colour deposited on the face of the fabric.

DISCUSSION

The experimental results indicate that migration occurs only when sufficient moisture is present. The amount required varies with the kind of textile fibres. It is very small with glass, larger with nylon, larger again with cotton, and larger still with wool and viscose. In all cases the figure at which migration ceases can be determined only within a range of a few per cent., within which the data agree with other data previously published on the drying of fibres.

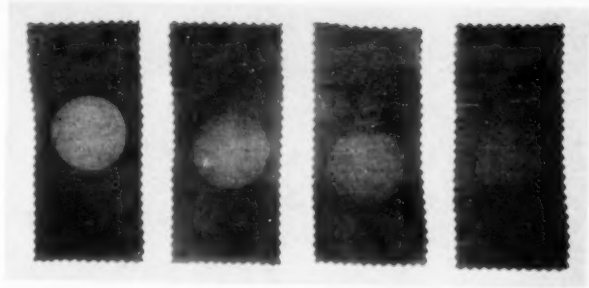
The centrifuge method[4,5] and the conductivity method[6] give data which put the fibres in the same order as that in the table above. The results of all methods are in reasonable numerical agreement for glass, nylon, cotton, and wool, but there is a discrepancy in the case of viscose rayon. This is possibly because of the sensitivity of the moisture absorption value for viscose rayon to the previous treatment the fibres have received.

Mechanical methods of removing water such as the ordinary preliminary operations of mangling, sucking, or centrifuging still leave a considerable amount of capillary water in the assemblies of fibres[4,5,7]. This water is in the capillary spaces between and around fibres, and can amount to as much as 20–30%[7]. Because of the numerous fibre-to-fibre contacts this capillary water forms a continuous film throughout the assembly of fibres and permits liquid migration to take place readily. As evaporative drying proceeds a stage is reached at



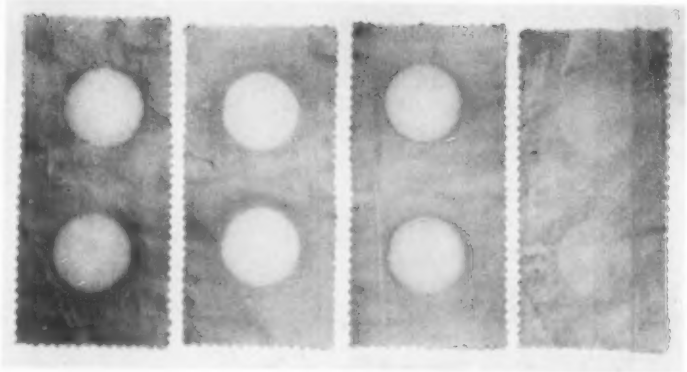
70% 58% 45% 34%

FIG. 1—Cotton



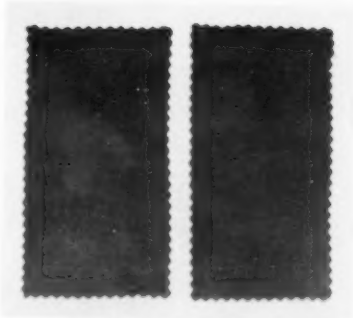
51% 62% 48% 42%

FIG. 3—Viscose



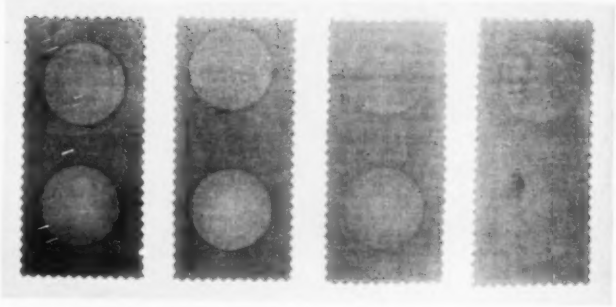
12% 9% 7% 3%

FIG. 2—Glass



41% 39%

FIG. 4—Viscose



31% 29% 27% 25%

FIG. 5—Cotton

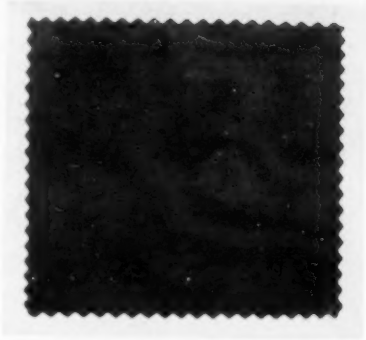


FIG. 7

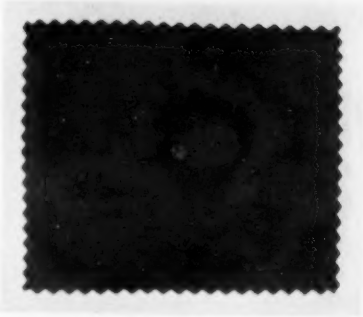
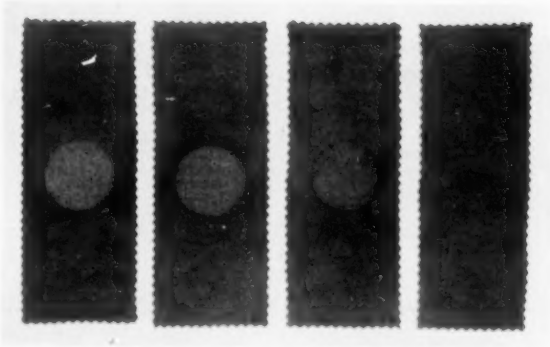


FIG. 9



46% 43% 39% 33%

FIG. 6—Wool

FIG. 8

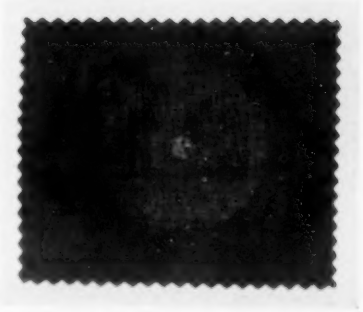


FIG. 10

which this capillary water has all vaporised, and there is then no continuous liquid phase through which migration can occur. When this stage is reached the remaining water is all or nearly all in the fibres. Any further drying reduces the area of water evaporating, since the surface of the fibres consists partly of fibre substance. At this stage there is a transition and a reduction in the area of water from that covering the whole surface of the fibres to the fraction of the surface of the fibres which is not fibre substance.

Partial covering of an evaporating surface or reduction of an available evaporating surface in various ways is known to reduce the rate of evaporation[8]. In drying this change is accompanied by a rise of temperature, as was previously reported[1]. Thus the cessation of migration and the second temperature step during drying correspond to approximately the same stage in the dehydration of fibres. After this stage the residual moisture is all or nearly all in the fibres.

In the migration experiments the phenomenon depends upon the presence of fibre-to-fibre contacts. It may be expected, and in fact it was observed, that closely constructed fabrics showed the effects of migration more distinctly than did more open ones.

The interesting migration phenomena shown in Fig. 7–10 exemplify how raising the temperature of part of the fabric transfers moisture via the vapour phase to another part, where the increased water content permits liquid migration effects to take place. This throws light on the observation that in high-temperature drying the migration effects were seen most clearly on the side further from the source of heat. It is now clear that in the early stages of drying there is a transient rise of water content on the cooler side. It illustrates also how difficult it is to avoid migration effects during heating or drying.

The experiment illustrated in Fig. 9 and 10 is particularly interesting. Here there has been a marked transfer of water vapour to the centre not in contact with the hot cylinder. The vapour has condensed there and on the underlying support, thereby raising the water content of the fabric to a sufficiently high level to give really marked migration of dye away from the centre. Evaporation of this migrating solution of dye when it reached dry fabric immediately in contact with the cylinder has deposited an intense ring of dye as seen in Fig. 9.

These experiments illustrate the ubiquity of liquid migration phenomena during heating or drying of fabrics and equally of other porous materials. Except for high-frequency heating or drying, where the heating is uniform[2], these effects cannot be avoided completely, but they can be minimised greatly. The more water which is removed mechanically and the lower the temperature of drying the less serious are the migration effects. Using solutes with affinity for the fibres or insolubilising the solute before drying reduces or even eliminates migration effects. Also, exposing the fabrics to the heating or drying conditions as uniformly as possible tends to reduce migration.

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Studies in the Fundamental Processes of Textile Printing
II—The Influence of Thickener Composition on Dye Transfer during Steaming

D. A. REILLY and H. A. TURNER

Previous work has been continued on the transfer of dye, during steaming, between a solid film of thickener substance and a film of regenerated cellulose in contact with it. Films were made from a variety of natural starches and other typical thickening agents. The general characteristics of dye transfer over the steaming period are similar for most of the thickeners examined, but the proportion of dye transferred at equilibrium may vary considerably between one thickener substance and another. The presence of appreciable numbers of anionic groups in the thickener molecule may lead to the rapid and complete transfer of dye to the cellulose. Possible relations between the chemical constitution of the thickener and the efficiency of dye transfer are discussed.

Introduction

GENERAL

In a previous paper[1], a new experimental procedure was described, by means of which might be studied the migration of water-soluble, substantive dyes from the film of thickener to the fibre substance in the steaming process for textile prints. This procedure was designed to reproduce the conditions which obtain during the technical process after the impression and drying of the printed fabric have been completed; variations were made in non-essential details in order that the dye transfer could be defined with sufficient accuracy. The study was confined to the transfer behaviour of the direct dyes from a starch thickener to cellulose. In this way, attention was concentrated on the diffusion of dye molecules into the cellulose during steaming. Other chemical mechanisms which

operate during the industrial steaming process whereby the colouring matter is made ready for diffusion, is formed *in situ*, or is fixed upon the fibre are thus excluded from direct attention.

In the present work, the same experimental procedure has been adopted without essential modification. A single pure direct dye, Chlorazol Sky Blue FF (*C.I.* 518), has been used throughout. The principal deviations from former conditions were necessitated by the differences in the mechanical properties of the films made from the respective thickening agents. Where possible, the thickeners were compared in films of the same thickness, so that equal initial dye concentrations corresponded to the same weight of dye opposite unit area of the surface of contact between thickener and cellulose phases. The steaming procedure and the method of estimation of dye in thickener or cellulose were the same as in the previous investigation.

COLOUR YIELD

As at present judged, this quantity depends entirely on visual impressions of the intensity of colour in a specified printed area. It is known that the same weight of a given dye may be applied to unit area of a cloth by one of the usual printing methods, and that, as conditions of impression, drying, steaming, and finishing change, so also may the final intensity of colour. A great variation may occur, *ceteris paribus*, when the thickener is changed, and this may be due to two principal causes—(*a*) differences in the distribution of dye within the thickness of the cloth, (*b*) differences in the final distribution of the dye between the applied thickener layer and the contiguous fibre substance. This distinction has already been discussed in the previous paper, and it has been shown that the present technique of investigation excludes, for all practical purposes, differences in the colour yield arising from the first cause, and tends to measure only the proportion of the total applied dye taken up by the fibre. It has also been pointed out that, with the carbohydrates and related substances as thickeners, essentially the same reaction as that which has been assumed to cause the fixation of direct dyes upon cellulose, and for which heats of reaction have been deduced for the combination of cellulose with specific direct dyes, can also be assumed to take place between dye and thickener molecules. This would lead in turn to the view that the ultimate equilibrium partition of dye between the two phases, thickener and cellulose, would be determined by the competing attraction of the two kinds of molecules for the molecules of the dye.

Furthermore, when the exhaustion at equilibrium is determined for a series of thickening agents, with the experimental conditions as nearly comparable as can be attained, it follows, since the cellulose phase is the same in each case, that the observed values will be in an inverted order of magnitude to the order of attraction of the respective thickening media for the dye. Using the word "affinity" in its more general sense, the lower the proportion of dye migrating to the cellulose, the greater the affinity of the thickener. A good deal of experimental evidence has been adduced for the view that fixation of direct dyes occurs through the formation of at least two hydrogen bonds between each dye molecule and suitably situated hydroxyl groups in the cellulose. If, for the sake of simplicity, the thickeners are considered first as pure carbohydrates, there should be the same basic reaction with the dye as for cellulose. The affinity of a specified thickener relative to that of cellulose, or the difference between that of one thickener and another, will then depend in each case upon the proportion of the total number of hydroxyl groups which, at the time of steaming, are accessible to dye molecules and are suitably disposed for reaction. This characteristic quantity for any film of thickener will depend upon a number of factors, including the degree of heterogeneity among the constituent molecules, the prevalence of branched or of straight chains, the arrangement of these molecules into close crystalline arrays or into looser amorphous structures, as well as the grosser structural characteristics of the films determined in part by the method of preparation. It is obvious that the present simple experimental technique will be unlikely to separate completely the contribution made by each of these factors to the final effect. The difficulties attending an accurate determination of the activity of a dye in a water-swollen fibre substance have proved, so far, to be the chief obstacle to the quantitative estimation of affinity by thermodynamic methods. A similar determination for dye in water-swollen thickener films is not likely to be less difficult.

In addition to this, however, the thickeners cannot be regarded as pure carbohydrates. In the starches there are present small but probably significant amounts of protein, phosphoric acid combined in different ways, and other sources of ionising groups, all of which may affect the rate and extent of dye migration. In the vegetable gums, uronic acids may constitute an important fraction of all the building units of the macromolecules. All these factors may need to be taken into account in interpreting the results.

RELATION TO ACTUAL PRINTING CONDITIONS

It has already been pointed out that the present investigation has to do with one set only of the factors which determine the contribution made by a given thickener to the final colour yield. A further warning must be issued against a too facile and direct interpretation of the results obtained under industrial conditions in terms of those obtained in the present work. With some exceptions, it has been necessary, in order to allow a fundamental comparison of the different thickeners to be attempted, that the proportion of dye to solid thickening should be kept approximately constant. This is, of course, wholly at variance with normal printing practice, where the proportion of thickener in a printing colour is adjusted, having regard to its own characteristics, to meet particular working requirements on the machine or screen. For a given depth of engraving, this means that an agent of high thickening power, used consequently at a low concentration, will dry down after impression to a comparatively thin film on the fibre, while one of low thickening power, used at greater concentration,

will dry down to a thicker film. The dye : thickener ratio will therefore be different in the two cases. It may be necessary at a later stage to compare thickening agents on this basis, but to do so initially would be to introduce unnecessary complication.

THICKENERS INVESTIGATED

Dye transfer to cellulose sheet (Cellophane 600 PT) from films of the following thickening agents has been measured—

Potato starch
Maize starch
Waxy maize starch
Hydrolysed maize starch
Oxidised maize starch
Commercial British gum
Gum tragacanth
Cellulose methyl ethyl ether (Cellose PC 450)
Sodium alginate
Starch carboxy ether product (Solvitose C)

In some of the experiments films prepared from mixtures of maize starch with sodium alginate in different proportions were also used.

Synopsis of Results

1. With purified Sky Blue FF, in the absence of added electrolyte, the curves relating time of steaming with (*a*) dye transferred to cellulose per unit area of surface of contact, (*b*) percentage of total dye transferred (exhaustion) are respectively similar in form for all the thickening agents except sodium alginate and Solvitose C. There is a continuous fall in the rate of transfer with time of steaming until an equilibrium value is reached.

2. The time required to reach equilibrium, and the extent of dye transfer at equilibrium, vary widely among the thickeners investigated.

3. The effect of a given change in the initial dye concentration in the thickener film upon the proportion of dye transferred at equilibrium to the cellulose is different for different thickening agents.

4. With as close an approach to identical conditions as experimental difficulties permit, the proportion of dye transferred at equilibrium from the respective thickener films increases in the following order—

Cellulose ether < Gum tragacanth < British gum < Potato starch < Normal maize starch < Waxy maize starch < Sodium alginate < Solvitose C.

5. It is difficult to prepare an acid-hydrolysed maize starch and then to remove all the added acid. If the acid is neutralised and the hydrolysed product is compared with unhydrolysed starch to which has been added the same proportion of salt as that formed during the neutralisation of acid in the hydrolysed product, hydrolysis appears to increase the exhaustion.

6. Increase in the concentration of dye in the film of thickener, obtained by applying the same weight to unit area of the surface of contact, but with the film diminished to half its thickness, causes a small but definite increase in the exhaustion.

7. The two thickening agents which contain a high proportion of ionisable (carboxyl) groups—sodium alginate and Solvitose C—each give ultimately complete exhaustion.

8. Admixture of a neutral thickening (maize starch) to the sodium alginate causes a diminution both in the rate of exhaustion and in the total exhaustion at equilibrium.

Experimental

MATERIALS

DYE—Chlorazol Sky Blue FF 200 (I.C.I.; *C.I.* 518) was purified in the usual way by the method of Mills and Robinson[2], being repeatedly precipitated from aqueous solution with sodium acetate, and the final precipitate then extracted with dry ethanol to remove the sodium acetate. The extracted material was dried overnight at 100°C. and preserved in a stoppered container. The purity was checked by colorimetric comparison with a sample of known purity in 20% aqueous pyridine.

SOLVENT FOR EXTRACTION—Commercially pure pyridine was distilled before use, and the fraction boiling at 112–115°C. retained. It was diluted before use with distilled water to a 20% solution (by volume), and preserved in a stoppered bottle away from the light.

METHYLENE BLUE (*C.I.* 922)—This was required for the determination of carboxyl groups in some of the films[3]. A specimen of commercial, zinc-free dye was purified by crystallising three times from hot aqueous solution. The purity was estimated by titration with titanous chloride solution standardised with Analar ferric ammonium sulphate. The product contained 95·0% anhydrous methylene blue ($C_{16}H_{20}N_3SCl$).

CELLULOSE FILM—Cellophane (600 PT grade) was steeped in five changes of distilled water over a period of two days. It was then preserved in water until required. The ash content was 0·172 g. per 100 g. dry cellulose. The mean thickness of the soaked film was 0·009 cm. The cellulose content of the wet sheet was determined by drying a specimen to constant weight at 110°C. and was 0·00475 g. per sq. cm.

POTATO STARCH—A high-grade commercial farina of low electrolyte content.

MAIZE STARCH—A sample of good-quality commercial corn starch supplied for textile printing.

WAXY MAIZE STARCH—A sample of authentic waxy maize starch for which we are indebted to the British Cotton Industry Research Association.

BRITISH GUM—A thin-boiling, fairly highly converted product deep yellow in colour and giving a dark brown solution.

GUM TRAGACANTH—A somewhat dark, lump printing gum ranging in colour from straw to light brown. As will be seen below, the ash content was high. Before use the gum was ground to a powder and mixed thoroughly.

CELLULOSE ETHER—Cellose PC 450 (Fine Dyestuffs & Chemicals Ltd.), a mixed methyl ethyl ether, in loose fibrous form, readily soluble in cold water to give a clear dispersion.

SODIUM ALGINATE—A foodstuff grade of high viscosity (Manucol *V*, Albright & Wilson Ltd.). It differs from most of the alginate products now

suggested for printing in that the viscosity has not been enhanced by the presence of a small proportion of the calcium salt.

SOLVITOSE C—This was received towards the end of the work through the courtesy of Messrs. W. A. Scholten's Chemische Fabrieken N.V. of Foxhol, Holland. It was used as received. Crystals formed when the film made from it was drying, pointing to the presence of electrolyte impurities. There has been no opportunity for a detailed purification or investigation of the product.

ASH CONTENTS OF THE THICKENERS—These were determined by ignition in the presence of concentrated sulphuric acid (Table I).

TABLE I

Thickener	Ash Content (g. per 100 g. air-dry material)
Potato starch	0·28
Maize starch	0·21
Gum tragacanth	3·55
Cellulose ether	0·29
British gum	0·40
Waxy maize starch	0·075
Sodium alginate	31·0

HYDROLYSED MAIZE STARCH—The maize starch mentioned above (5 g.) was stirred into 100 ml. water as in the normal method for making a paste (see below), using the apparatus described in a previous paper[1]. The water in the surrounding bath was initially at room temperature, and was brought slowly to the boil, with continuous slow stirring of the suspension. When the bath reached the boil, 5 ml. of 0·167 N. sulphuric acid was added. After 25 min., with the bath at the boil, 5 ml. of 0·167 N. caustic soda was added, and cooking continued for a further 5 min. The strength of the solutions of acid and alkali was so chosen that the concentration of sodium sulphate in the prepared dry film would be approximately the same as the concentration of dye. The dispersion after hydrolysis remained liquid at room temperature.

OXIDISED MAIZE STARCH—Maize starch (200 g.) was suspended by vigorous stirring in 6 litres of sodium hypochlorite solution (30 g. available chlorine per litre) at room temperature. After 90 min., the suspended starch grains were filtered off, and washed thoroughly with distilled water until the washings gave no reaction with either silver nitrate or starch–iodide. After removal of as much water as possible on the filter, the product was dried overnight at 30–40°C. The treatment had caused a marked decrease in viscosity.

PREPARATION OF FILMS

The preparation of solid films of potato starch containing direct dye has already been described in detail[1]. This procedure was adhered to as far as possible in the preparation of films from other materials, but the consistency of the paste and the strength and adhesion to glass of the film made from it differed in some cases so widely from the corresponding properties in potato starch that some changes in detail were necessary.

POTATO STARCH—It is unnecessary to repeat the description of film making. As in previous work, the thickness of the spread layer of 10% paste was 0·165 cm.

MAIZE STARCH—Here, a 10% paste is inconveniently viscous for spreading, even at the boil, so that one of 5% was made and spread in a layer of double thickness. As the viscosity of a 5% (50 g./litre) paste is rather low when hot, the rise in viscosity when the granules burst during cooking was difficult to observe. To ensure adequate cooking, the starch suspension was placed in the apparatus with the water in the bath cold. This was brought slowly to the boil, and cooking continued for 30 min. after this point had been reached. The starch paste tends to form a hard crust at the top edge of contact with the beaker. It was therefore filtered rapidly through a sieve of copper gauze immediately before spreading. The concentration of thickener and dye in this and other pastes tended to be somewhat irregular from one preparation to another. Just before the end of the preparation, therefore, the paste was weighed and any deficiency from the original total weight adjusted by the addition of water. This improved greatly the accurate reproducibility of the film. As noted above, the paste was spread in a layer of twice the normal thickness (i.e. of 0·33 cm.), by putting two glass strips one on top of the other at each edge of the glass spreading plate. The dye concentration was half that for a normal 10% starch paste.

The film was dried in the apparatus used in the previous work, except that drying was found to be too slow in an atmosphere of 75% R.H. The relative humidity was fixed at 65%, using saturated ammonium nitrate as conditioning agent. The films were more brittle than those of potato starch, so that increased care was needed in removing them from the plate and in cutting discs from them. Occasionally it was necessary to condition them at 100% R.H. for a short time before they could be handled. They were preserved in an atmosphere at 75% R.H.

WAXY MAIZE STARCH—Procedure was generally the same as for normal maize starch. The films do not form skins or crusts, so that filtration is unnecessary. The paste sets very rapidly on cooling, so that it is spread as hot as possible on previously warmed glass plates.

HYDROLYSED MAIZE STARCH—Hydrolysis took place during cooking, and the preparation of a 5% paste has therefore been described above. The viscosity of the paste is very low, and ordinary knife spreading is impossible, so that the film was made by casting. The usual trough-like plate was converted into a shallow box by closing the two open ends with glass strips and sealing the joints with cellulose acetate cement. The plates were carefully levelled inside the drying box, and the paste poured in through a gauze filter. It was finally levelled off with a knife as in the spreading procedure, and drying at once commenced, taking care to avoid disturbance until solidification had taken place. The films were less brittle than those of the unhydrolysed starch.

OXIDISED MAIZE STARCH—The oxidation has already been described. From the dry product, a

5% paste was prepared. It was fluid in the cold, and was cast into films in the same way as the hydrolysed maize starch.

GUM TRAGACANTH—With this product long soaking and thorough cooking are necessary. Filtration is also necessary to remove insoluble material. The adhesion of the dried films to glass is considerable, and since the films are brittle when fully dry, and very plastic when their moisture content has been increased to the point at which they can be detached from the glass, some modification of the normal method of film making is required. The procedure finally adopted is outlined as follows—

(i) Powdered gum (9 g.) soaked overnight in 150 ml. distilled water

(ii) Suspension cooked for 2 hr. in the standard apparatus, water being added from time to time to make up for evaporation

(iii) Paste strained through cloth, loss in weight being noted

(iv) Dye added as calculated volume of a standard solution; cooking resumed for a further period until the total weight, diminished by evaporation, corresponded to a 6% concentration of thickener

(v) Air bubbles removed by placing in vacuum desiccator and cautiously reducing pressure, avoiding priming

(vi) Spread hot to a depth of 0·3 cm.

(vii) Dried in moist air (75% R.H.) in usual way

(viii) Conditioned at 100% R.H. for 2 hr.; brought into open air, and moisture content allowed to fall until the film could be removed without breakage or distortion

(ix) Preserved at 75% R.H.

BRITISH GUM—This gave a paste of low viscosity, and it was therefore prepared at twice the normal strength (200 g./litre) and spread to a thickness of 0·075 cm., with twice the normal concentration of dye. No difficulty was encountered in making the paste by the normal method. The dried films adhered very strongly to the glass, and, after being conditioned over water for 1 hr., they were removed from the plate in narrow strips.

CELLOSE PC 450—This is soluble in cold, insoluble in hot, water. The requisite weight of the ether was stirred into boiling water, and stirring was continued while the water was boiled for some minutes with a direct flame in order to distribute the light fibrous material well and to remove entangled air bubbles. The jacket, containing boiling water, was then put into place, the standard dye solution added, the total volume adjusted with distilled water, and the whole apparatus left to cool slowly with uninterrupted stirring. As cooling continued, the ether gradually dispersed. An apparently homogeneous, air-free paste resulted. The paste was made at a concentration of 3·3% of thickener and spread in a layer 0·45 cm. thick. It spread easily, and after drying at 75% R.H., a strong, readily detachable film resulted.

SODIUM ALGINATE—This material swells fairly rapidly in water, and care is required to avoid the formation of lumps. The Manucol *V* (5 g.) was added, little by little, to 100 ml. of water at the boil, stirring the powder in by hand and pressing out any lumps that formed. After the addition of dye, the paste was allowed to cool with continuous slow stirring in the paste-making apparatus. The final volume was adjusted with distilled water. The dispersion was fluid at room temperature, and it was therefore cast in a shallow box as with hydrolysed maize. The dispersion was spread to a thickness of 0·15 cm., giving a dry film of half the normal thickness. The films were fairly strong and pliable, and could be removed from glass without great difficulty.

SODIUM ALGINATE–MAIZE STARCH MIXTURES—Two mixtures were made, containing, respectively, 80% and 50% alginate. A 5% alginate paste (100 g.), cold and containing the necessary weight of dye, was stirred slowly and continuously while the requisite quantity of hot 5% maize paste was added slowly through a wire sieve. After thorough mixing, the paste was spread to a thickness of 0·15 cm. in the usual way. The dried films containing 20% maize resembled alginate films in strength and flexibility; those containing 50% maize were distinctly more brittle.

ATTACHMENT OF FILMS TO CELLULOSE

The cutting of discs from the dry thickener films and their attachment to cellulose sheet, worked out for potato starch and previously described[1], required some modification for films of some other thickeners, generally because perfect adhesion was less readily obtained.

POTATO STARCH—No further modifications were introduced in the procedure for cutting the discs, checking the dye content, combining with soaked Cellophane strips, and steaming.

MAIZE STARCH—The same procedure was adopted as for potato starch. Maize starch film was very prone to a difficulty encountered less frequently with potato, viz. the formation during the steaming of small bubbles between the starch and the cellulose film. This caused areas of separation between the two films at which dye transfer was impossible. Means to avoid this trouble were sought without great success, and finally a larger number of determinations were made, the results from imperfect ones being rejected.

HYDROLYSED MAIZE AND WAXY MAIZE STARCH—These were combined and steamed as for normal maize starch. The proportion of faulty results was fairly large.

GUM TRAGACANTH—Films of this material become too soft on contact with water-soaked cellulose sheet for combining to be successful by the normal procedure. After a number of trials, the following method was adopted. All loose water was removed from the strip of Cellophane with filter paper. A strip of tragacanth film, rather less in width than the Cellophane, was cut and was then passed, along with the Cellophane, through the nip of a small mangle with soft rubber-covered rollers. Light pressure only was needed, and the two strips were allowed to touch each other only in the nip, otherwise creases formed in one or the other. The combined strips were steamed together, and after

removal from the steamer, the usual discs of $\frac{11}{16}$ in. diameter were cut for dye estimation.

BRITISH GUM—This was treated in the same way as tragacanth. Since dry films are very brittle and damp ones very soft, combining followed a careful conditioning in an atmosphere of 97% R.H.

OXIDISED MAIZE—The films were not difficult to handle, but liquefied completely on steaming, so that no dye transfer could be obtained.

CELLULOSE ETHER—Although adhesion to cellulose was good in the cold, the films fell apart on steaming, presumably because of the low solubility of the ether at the higher temperature. Finally the ether was combined with Cellophane in the same way as tragacanth, and the compound strip tightly wrapped, Cellophane outwards, round the outside of a short length of glass tube, 0·5 in. in diameter, the ends being secured with a bulldog clip. The whole was hung in the steamer by means of the clip, with the tube horizontal. Regular but very low transfer of dye took place on steaming, and it was thought that the two films might not be in thorough contact. Accordingly, a weight of 100 g. was hung to the tube by a wire stirrup, so that the pressure of contact would be increased. Since there was no corresponding increase in the dye transfer, it was concluded that measurements made in this way did in fact represent the true transfer properties of the film.

SODIUM ALGINATE—Neither the squeegee method nor the mangle method was successful. Finally, strips of alginate were attached to the Cellophane by hand, and then clipped round a tube for steaming as with the cellulose ether. This gave complete and regular transfer, and it was also adopted when films from the maize–alginate mixture were steamed.

STEAMING

No change was made in the steaming technique.

SEPARATION OF FILMS AFTER STEAMING

POTATO AND MAIZE STARCH—After removal from the steamer and cooling for a few minutes, it was usually possible to remove the starch film intact or in a few pieces. The dyed areas of the cellulose were then examined against the light, when any traces of starch remaining showed up as dull patches. They were removed by wiping with a damp cloth.

TRAGACANTH, BRITISH GUM, HYDROLYSED MAIZE STARCH—Films of these materials absorb more moisture and are softer than those above. After a number of trials they were removed as follows. After steaming, the strips were placed at once, thickener side down, on a sheet of moderately absorbent paper (duplicating paper). On carefully peeling away the Cellophane, most of the thickener remained on the paper. Any not removed was wiped from the Cellophane with a damp cloth.

CELLULOSE ETHER—On removal from contact with the glass tube, the two films separated spontaneously.

SODIUM ALGINATE, WAXY MAIZE STARCH—These films were so soft after steaming that the only satisfactory method was to wipe away the whole film with a damp cloth.

COLORIMETRIC ESTIMATION OF DYE IN CELLULOSE

The methods already described, in which transferred dye was extracted from the cellulose (and, on occasions, also from the thickener film) with 20% aqueous pyridine, and the concentration of dye in the extract measured in the Spekker photoelectric absorptiometer, were used without modification.

THICKENER CONTENT OF DISCS

This was measured by direct weighing of a number of the discs, or of other known area of the film, after conditioning at 75% R.H. A useful preliminary check for serious deviation from the mean value was given by measurements of the thickness with a Schopper micrometer gauge.

ASH CONTENT OF THICKENERS

No detailed analysis of the thickening materials was undertaken. As a guide to the maximum possible electrolyte content, ash values were determined. It will be seen from Table I that all the natural thickeners except gum tragacanth and sodium alginate had low values. The determination was made by burning off a weighed sample of the material in a silica crucible, moistening the residue with concentrated sulphuric acid, and igniting to constant weight at a dull red heat.

METHYLENE BLUE ABSORPTION

Because of the unusually high transfer from sodium alginate, it was thought advisable to attempt a determination of carboxyl value in some of the other films. These determinations could not be made with some of the more soluble films. The method described by Davidson and Nevell[3] for carboxyl determination with normal and modified celluloses was adopted, and the original paper should be consulted for full details of its development. The weight of the material used was that which gave exactly 50% exhaustion of a solution containing 0·673 g. (2·0 millimoles) of purified (95%) methylene blue hydrochloride per litre buffered with 1·563 g. (6·25 millimoles) of sodium diethylbarbiturate per litre.

Preliminary experiments were made in which different weighed amounts of potato starch film (moisture content 18·3%) were each placed in a 150-ml. stoppered flask, covered with 25 ml. of the buffered dye solution, and allowed to stand at room temperature until complete absorption has taken place. The concentration of the solution was then determined in the Spekker. It was found that 0·1048 g. of film gave 50% exhaustion, corresponding to 2·92 milliequivalents of carboxyl per 100 g. dry starch. The corresponding value for unmodified maize starch films was 2·14. The full validity of this method depends on there being no dissolution of the film in the dye solution during the determination. To test this, the weight of the dyed sample after the experiment was compared with the original weight (Table II). The increase of weight is greater than that of the dye absorbed, and corresponds almost certainly to the inclusion of some of the buffer. These figures do not, therefore, prove conclusively that no dissolution takes place, but they show that this loss cannot be large and

TABLE II

	Potato Starch	Maize Starch
*Dry weight, original ...	(a) 0·0763	0·1208
	(b) 0·0985	
Dry weight, after absorption	(a) 0·0820	0·1246
	(b) 0·1050	

* Moisture content determined on separate samples by drying to constant weight at 110°C.

that the results are of the correct order. In any case the carboxyl content is low for each material. Carboxyl determinations could not be made with tragacanth, hydrolysed maize starch, British gum, cellulose ether, or sodium alginate. Although they would have been of great interest the films were too soluble. With sodium alginate the carboxyl value is of a completely different order; calculating from the accepted constitution, it is 570 milliequiv. per 100 g., or some 250 times that of the starches.

drying to constant weight. Suspension of the thickener films alone in the steamer was unsatisfactory, since many were so soft that they either broke during steaming or could not be removed quickly from the suspending hook. Finally, a determination was made by supporting them on a film of cellulose, the moisture content of which was found in separate control experiments. Unfortunately the variation in the results with the same film material in different experiments was so great that no significant information could be obtained. This problem has since received further attention, and it has been found that much more elaborate procedures will be necessary before accurate values can be obtained.

EXPERIMENTAL RESULTS

The principal details of the films are set out in Table III, which also includes references to the

TABLE III

Thickening Agent	Mean Thickness of Film (cm.)	Mean Wt. of Disc (g.)	Ash Content (%)	Dye Content of Film (mg. per disc)	Dye Content of Film (g. per 100 g. dry film)	Equilibrium Exhaustion (%)	Transfer Curve (Fig.)
Potato starch	0·013	0·030	0·28	0·0580	0·190	68	1
				0·1075	0·355	54	1
				0·1550	0·516	63	1
Maize starch	0·016	0·039	0·21	0·1080	0·282	57	2
				0·1790	0·468	70	2
	0·0075	0·019	0·21	0·1190	0·626	61·5	3
				0·1680	0·884	72	3
Waxy maize starch	0·0075	0·018	0·075	0·1020	0·566	63	4
Unhydrolysed maize starch + Na_2SO_4	0·016	0·038	0·21	0·0495	0·130	49·5	5
			(+1·2% Na_2SO_4)				
				0·1040	0·238	71	5
Hydrolysed maize starch + Na_2SO_4	0·016	0·038	0·21	0·0475	0·125	64	6
			(+1·2% Na_2SO_4)				
				0·1160	0·305	78	6
British gum	0·017	0·038	0·040	0·1660	0·447	33·5	7
				0·2955	0·778	35·5	7
Gum tragacanth	0·016	0·037	3·55	0·0805	0·217	40·5	8
				0·1625	0·439	33	8
				0·2400	0·649	33·5	8
Cellose PC 450	0·017	0·043	0·29	0·1350	0·314	18	9
				0·1850	0·430	23	9
Sodium alginate	0·008	0·020	31·0	0·0740	0·370	100	10
				0·1600	0·800	100	10, 11
Sodium alginate + Maize starch—							
80 : 20	0·0075	0·018	—	0·1320	0·733	98	11
50 : 50	0·0080	0·020	—	0·1600	0·800	87·5	11

ABSORPTION OF WATER BY THE THICKENER DURING STEAMING

Results obtained by Munshi[1], and from later unpublished work[4], suggest that the extent of dye transfer is related to the proportion of water present in the system during steaming, and, particularly, to the ratio in which it is distributed between the thickener and the cellulose phases. Preliminary experiments were carried out in an attempt to determine the water content of the respective thickener films during steaming by the simple procedure of removing them as rapidly as possible from the steamer, and transferring immediately to a stoppered weighing bottle. The moisture content could then be determined in the normal way by

figures containing the transfer curves of Sky Blue FF from the respective films to cellulose on steaming. In these curves, the exhaustion of the thickener film, i.e. the percentage of the dye originally present in the film which has migrated to the cellulose, is plotted against the time of steaming. In some of these experiments, the films of a thickening, such as maize starch, are employed in two thicknesses, as already noted, and in more than one concentration. In this way, a comparison may be attempted with the films of other thickening materials for which the properties of the dispersion make it difficult to prepare other than quite thin films.

Most of the individual curves are self-explanatory, and little detailed comment seems to be required. Where marked deviations occur from a normal dyeing curve, it is found invariably that there are unusual difficulties in obtaining uniform adhesion to the cellulose film, or in avoiding bubble formation during steaming.

The opportunity has been taken of comparing on a single graph the results for all the thickeners in films where the dye concentrations are approximately the same (Fig. 12).

Discussion

COMPARISON OF DIFFERENT THICKENERS

From Fig. 12, covering the whole scope of the present work, the most striking observation is that of the large differences in dye transfer at equilibrium among the films of the different thickeners. At the same time, from the details of the experimental work, and especially because of the different expedients that have been found necessary in the preparation of films of the respective thickeners, the conclusion has been strengthened that the present investigation of this complex subject must be regarded mainly as serving to define the methods for a much more thorough and prolonged attack. It is not easy to make films from a representative variety of thickening agents, each of a standard thickness and dye concentration. It has been shown also that the problem of ensuring efficient contact with the cellulose film varies in detail with almost every agent examined. Work on dyes of low substantivity (levelling acid dyes, Indigosols), to be described in a later paper, has yielded results which suggest that the distribution of water in the cellulose–thickener system, before and during steaming, has an important influence on the transfer behaviour. It is not to be expected, therefore, that anything more than an approximate comparison can be drawn between, say, the present results with maize starch, where the side of the film not in contact with cellulose is freely accessible to the steam, and those with tragacanth, where this side is in contact with glass.

The strongest indication of the necessity for a very long and detailed investigation is obtained when the relations between equilibrium exhaustion and initial concentration of dye in the thickener film are considered. It has already been shown, for Chlorazol Sky Blue FF in potato starch[1], that this relation is not a simple one. In view of the possibility of different complex relations, on the one hand for the same dye in a number of thickening agents, and on the other for different dyes with each thickener, it is difficult, until the survey has been much extended, to decide on an effective basis for comparison of one thickener with another. Should they all be compared with each other at a standard dye : thickener ratio in films of a standard thickness, or should some critical point, of possible theoretical significance, be sought on the curve relating equilibrium exhaustion with initial dye concentration for each thickener, and the comparison carried out under conditions represented by this point? From considerations of this kind, it was decided to give an account of what had been achieved to date rather than to delay until results had accumulated which might allow the extended comparison to be accomplished satisfactorily.

REPRODUCIBILITY OF RESULTS

Where, as with potato starch, it has been possible to define the conditions simply and with reasonable accuracy, and to obtain a smooth transfer, the reproducibility is shown to be good in the results obtained by different workers with completely independent film preparations (Table IV).

TABLE IV

	Mean Initial Dye Concn. (mg. per disc)	Equilibrium Transfer to Cellulose (mg. per disc)
Series A ...	0·1550	0·099
Series B ...	0·1580	0·096

DIFFERENCES BETWEEN EXHAUSTIONS WITH DIFFERENT THICKENERS

It has already been noted that, in the absence of added electrolyte, and with potato starch which has a very low electrolyte content, the exhaustion of pure Sky Blue FF from thickener films is considerable. (When this dye is applied from solutions of the concentrations normally used in dyeing, the exhaustion is negligibly small.) This behaviour is now confirmed to a greater or less extent with other starch and modified starch films, although the specific exhaustion does vary from one film material to another. The general high exhaustion has been attributed to two factors—(*a*) the use of a high dye concentration in a phase external to the cellulose of such small volume that this concentration can be attained with a relatively low dye : cellulose ratio, i.e. the initial concentration of the dye and therefore of its accompanying sodium ions is of the same order in the "dyebath" as the final concentration in the fibre substance; (*b*) the diminution of the negative potential at the cellulose surface— which impedes transfer of dye to the cellulose— as compared with that which is established when an aqueous dye solution is used. The possibility of chemical attachment of dye molecules to the thickener molecules has been mentioned as a factor opposing migration to the cellulose phase. With these three factors in mind, it is possible to suggest three general reasons for different equilibrium exhaustion values with different thickening agents in systems where constant weights of thickener, cellulose, and dye respectively are associated with a standard area of contact between thickener and cellulose films—

(i) The different films have different internal structures, and during steaming swell and take up water to different extents. With constant weight of dye this implies different dye concentrations external to the cellulose.

(ii) The interphase potential differences are different from one thickener to another.

(iii) The attachment between molecules of the dye and those of the thickener is almost certain to vary widely among films of different chemical constitution and of different internal structures.

All these factors should ultimately be capable of direct experimental assessment, but our experiences

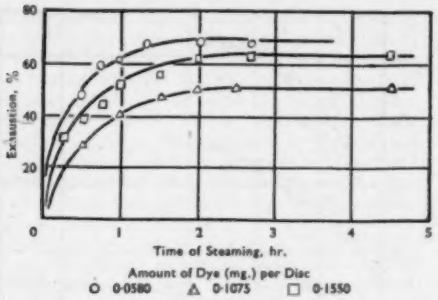

FIG. 1—Transfer of Chlorazol Sky Blue FF from Potato Starch

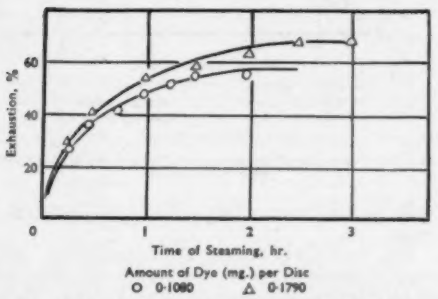

FIG. 2—Transfer of Chlorazol Sky Blue FF from Maize Starch

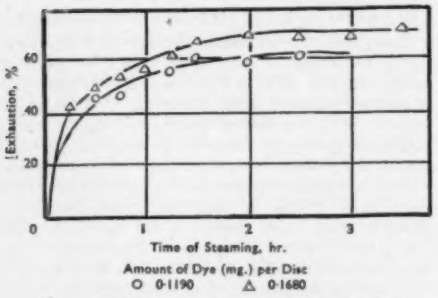

FIG. 3—Transfer of Chlorazol Sky Blue FF from Ordinary Maize Starch in Films of Half Normal Thickness

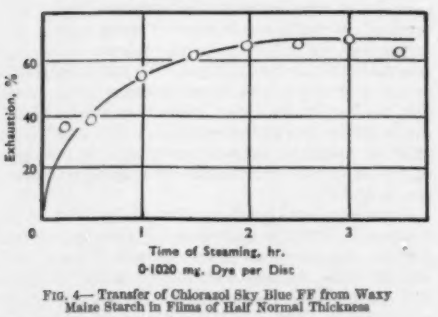

FIG. 4—Transfer of Chlorazol Sky Blue FF from Waxy Maize Starch in Films of Half Normal Thickness

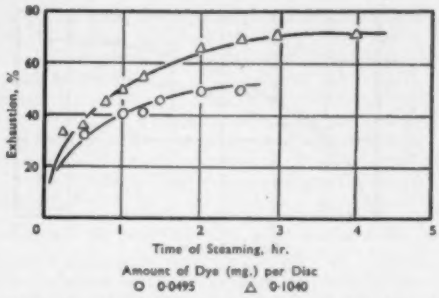

FIG. 5—Transfer of Chlorazol Sky Blue FF from Unhydrolysed Maize Starch plus Sodium Sulphate

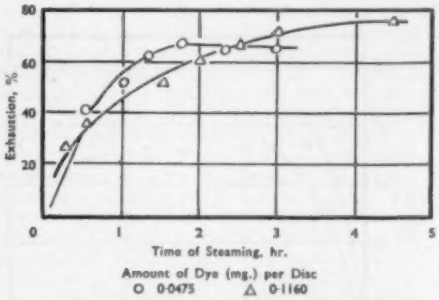

FIG. 6—Transfer of Chlorazol Sky Blue FF from Hydrolysed Maize Starch plus Sodium Sulphate

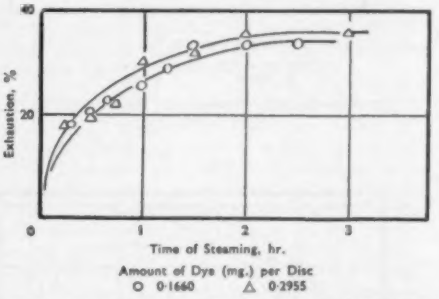

FIG. 7—Transfer of Chlorazol Sky Blue FF from British Gum

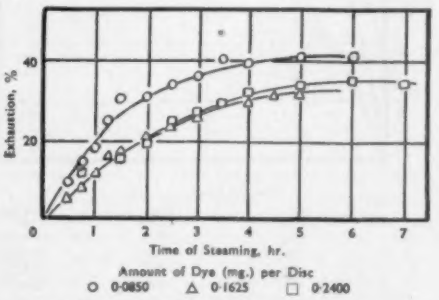

FIG. 8—Transfer of Chlorazol Sky Blue FF from Gum Tragacanth

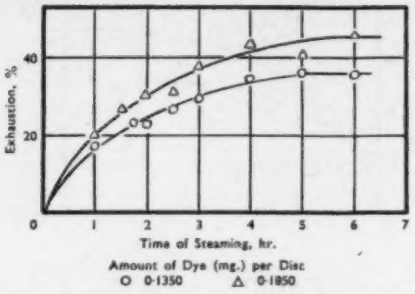

FIG. 9—Transfer of Chlorazol Sky Blue FF from Cellose PC 450

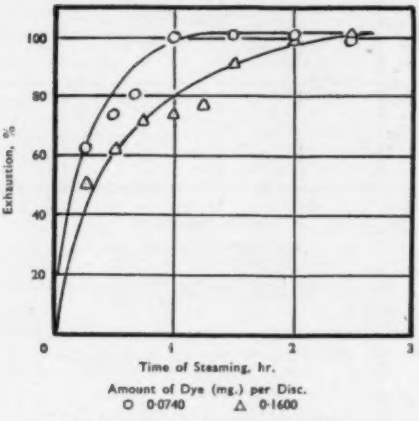

FIG. 10—Transfer of Chlorazol Sky Blue FF from Sodium Alginate

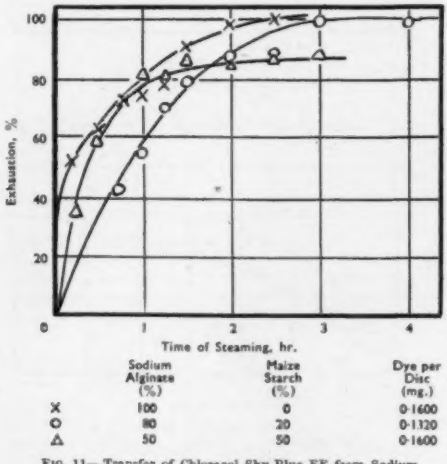

FIG. 11—Transfer of Chlorazol Sky Blue FF from Sodium Alginate–Maize Starch Mixtures

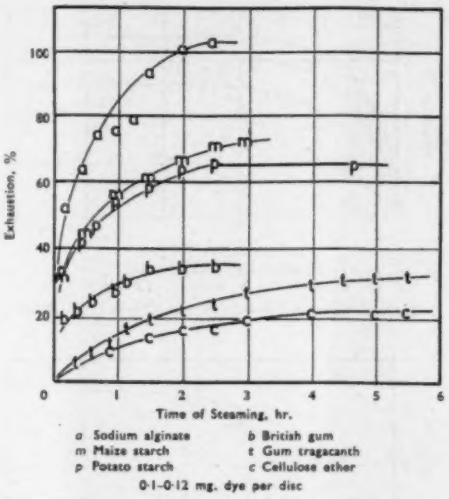

FIG. 12—Comparison of Exhaustion of Chlorazol Sky Blue FF with Different Thickeners

with (i) have shown that considerable difficulties exist.

The results which show the simultaneous operation of factors (i) and (ii) most clearly were obtained in experiments with sodium alginate films. These results were mentioned briefly in a previous paper[1], and will now be discussed in greater detail.

DYE TRANSFER FROM FILMS OF SODIUM ALGINATE

Even with the difficulties experienced in obtaining satisfactory adhesion, it will be seen on comparing Fig. 10 with Fig. 1–9 that the exhaustion of sodium alginate films is complete. It is unfortunate that these difficulties have brought about a scatter of points on the curves and this does not render deductions from the shape very profitable. About the ultimate attainment of 100% exhaustion there is no doubt; at the end of two hours it is difficult to see any colour in the thickener film. The possible mode of action of the thickener can be considered in different ways. As a surface effect, the high concentration of ionised carboxyl groups can give rise to a preponderating negative charge on the thickener side of the interphase boundary, completely eliminating the original "potential barrier" of cellulose in contact with water, and acting also as a barrier to the reverse process of desorption from the cellulose. With this valve-like action, not only is a much greater proportion of the dye molecules initially present in the alginate available for dyeing the cellulose, but it would seem that the migration may be encouraged by repulsion of dye ions into the cellulose after they have passed the boundary.

Considered as an effect through the bulk of the thickener film, the presence of carboxyl groups implies the presence of an equivalent number of sodium ions. Initially, therefore, in the thickener film, the sodium counter-ions of the carboxyl groups are added to those of the dye molecules themselves

and the already high concentration of sodium ions in the film are increased. The cellulose is virtually free from diffusible cations at the beginning, so that diffusion into the cellulose of sodium ions, accompanied by dye ions, is further encouraged. The effect of the sodium ions of the alginate is similar to that produced by the addition of, say, sodium chloride to a film of neutral thickening material. It is the inverse of the effect on dye absorption produced by the formation, through oxidation, of carboxyl groups in cellulose. Also, for an equivalent number of sodium ions, sodium carboxylate groups should be more efficient in encouraging diffusion into the cellulose than sodium chloride, since the sodium ions are attached to non-diffusible anions, and cannot migrate to the cellulose during steaming. They are unable, therefore, to reduce the difference in sodium ion concentration between the two phases as steaming progresses.

This deduction is confirmed by comparing the present results with those obtained by Munshi and Turner[1] for addition of sodium chloride to potato starch films. They found, for films containing 0·091 mg. pure Sky Blue FF, in potato starch, an equilibrium exhaustion of 69%. The addition of a weight of sodium chloride equal to the weight of dye present (mol. wt. dye : mol. wt. NaCl = 16 : 1) increased the equilibrium exhaustion to 87%, five times this weight of salt to 93%, while a tenfold addition caused no further measurable increase in the exhaustion.

There is one Na^+ ion for each 248 units of molecular weight in a molecule of Sky Blue FF and one for each 198 units in a sodium anhydromannuronate group. The alginate is thus up to 25% more effective, weight for weight, than the dye in providing Na^+. As, however, there is only 3 g. of dye present per 1000 g. of alginate, the latter is able to supply up to 400 times the number of sodium ions as compared with the dye, and this concentration should not be diminished through diffusion of Na^+ ions during steaming. We have seen that the maximum equilibrium exhaustion in Munshi and Turner's experiments was reached when the added electrolyte could supply sodium ions equal to approx. 20 times those supplied by the dye molecules at the beginning of the experiment. The extraordinary effect of the high concentration of non-diffusible sodium ion in the thickener thus seems to be demonstrated very strikingly.

It is interesting to compare the values for carboxyl content given by methylene blue absorption determinations on starch with those for sodium alginate. They are maize 2·14 and potato 2·92 milliequivalents per 100 g. respectively. The formula value for sodium alginate is 556 milliequivalents per 100 g. The effect of the carboxyl groups in the starch is therefore negligible in comparison.

That the argument, developed above to explain the very high exhaustion from sodium alginate compared with that of starch, is not wholly satisfactory is shown when the results with films containing mixtures of maize starch and sodium alginate are considered. With a film containing 50% of alginate the equilibrium exhaustion has dropped to 87%, although the supply of sodium ions is still ten times greater than with the additions of sodium chloride to potato starch which give exhaustions of 93%. It is obvious, therefore, that other factors, e.g. the effects of swelling, difference in internal structure with the films of different materials, and, perhaps also, a stronger attachment of dye molecules to starch than to alginate, are all operating.

AFFINITY OF THICKENER MATERIAL FOR DYE

In this discussion, the term "affinity" is used in the popular and qualitative sense and not in the sense employed by Meggy[5], Marshall and Peters[6], and others, where it denotes the standard free energy change when dye is adsorbed from aqueous solution by a fibre substance. To calculate the affinity it is necessary to determine the activities of the dye ions both in water and in cellulose. For comparatively dilute dye solutions the problem is simplified, since ion concentrations may be taken as equal to the respective ion activities. The activities in the solid phase are more difficult to determine and require the use of arbitrarily selected constants. In the present case similar constants would be required also to evaluate the ion activities in the thickener phase. There would therefore be a constant, the value of which could not be determined directly, in both thermodynamic potential terms of the affinity equation instead of in one only. It is not possible to simplify the problem by proceeding in two stages and determining first the affinity of the dye in starch films relative to water by dyeing these from aqueous solutions, since the film dissolves in water at 100°C.

The results with two thickeners not derived from starch may first be commented on briefly.

EXHAUSTIONS WITH CELLULOSE ETHER AND WITH GUM TRAGACANTH

The equilibrium exhaustion value for methyl ethyl cellulose is low, and this result is not unexpected. Three possibilities present themselves— (*a*) The insolubility of the film at 100°C. suggests that insufficient water may be present in it, during steaming, to dissolve much of the dye present, and therefore the overall mobility of the dye will be low. (*b*) The unfavourable conditions suggested in (*a*) may be aggravated by the experimental necessity of keeping the outer side of the thickener film in contact with glass, and not fully accessible to steam. (*c*) The low etherification value necessary to obtain water solubility in the product renders it likely that large regions in the internal surface of the film will be equivalent to adsorbing surfaces of cellulose itself, giving an affinity equivalent to that which obtains in the Cellophane, and much greater than that of the starch thickeners.

The low values for exhaustion from tragacanth are much more difficult to explain without further precise knowledge of the structure of the films it produces. The molecules of this gum have been shown to include uronic acid residues[7,8]. It has a high mineral content. The disadvantages of steaming with the outer surface in contact with glass have already been commented upon. The low

results with this gum and with cellulose ether suggest that results with locust-bean gum, which has a low solubility at steaming temperatures, and with a readily soluble gum like arabic would be of interest.

EXHAUSTION WITH DIFFERENT VARIETIES OF STARCH

It is probable that the effects of constitution and structure of films from different varieties of starch upon the rate and extent of dye transfer may resolve themselves for the purposes of study into two groups—(*a*) effects of the gross structure of very heterogeneous film material in which vestiges of the original granules are embedded in more completely dispersed starch, (*b*) effects attributable to the relative proportions of amylose and amylopectin in starches of different natural origins. These differences are, of course, of great importance in technical printing itself, influencing as they do the mechanical properties of the colour paste, which in turn affect the quality of the impression in respect of levelness and penetration. In these experiments such effects are excluded almost completely, and attention is directed more particularly to the number and size of the open spaces in the film through which water can penetrate and facilitate the dissolution and diffusion of the dye, and the arrangement of the hydroxyl groups on the walls of these spaces, which can affect the attachment of dye molecules. It has already been suggested that in cellulose and in the different varieties of starch the chemical reaction between dye and starch is likely to be the same, viz. hydrogen bond formation at hydroxyl groups in the carbohydrate, and that for effective attachment there must be two such bonds for each dye molecule. The difference in affinity between one carbohydrate polymer film and another should therefore be related to the number of pairs of hydroxyl groups in each film which are accessible to the dye and are spaced at the correct distance for interaction in the manner suggested.

Studies of the properties of the two most sharply differentiated fractions of natural starch—amylose and amylopectin—are numerous[9–22], and the question is related to the swelling behaviour of the granules, since there is evidence that these fractions occur in different relative amounts in different parts of the granule. The most important features which seem to be generally accepted are—

(i) AMYLOSE—Predominantly an unbranched chain molecule of α-glucoside units leading to marked tendency towards crystallisation, with the associated properties of low solubility in water or in mixtures of water and aliphatic alcohols, and the tendency to be adsorbed on cellulose surfaces. It is largely responsible for the retrogradation of starch pastes, in which sparingly soluble crystalline material separates out on standing. In potato starch, which contains the highest proportion of ester-combined phosphoric acid, it is lower in phosphorus content. It forms compact but inelastic films.

(ii) AMYLOPECTIN—A ramified, or laminated, molecule consisting primarily of α-glucoside units. It disperses readily in water, giving stable dispersions with little tendency towards retrogradation or surface-film formation on cooling. It gives films of great swelling capacity, soft and pliable. It is mainly responsible for the formation of gels in starch pastes, since amylose when brought into solution in hot water separates in "short" or powdery solid forms on cooling. The combined phosphoric acid of potato starch seems largely to be present in the amylopectin fractions.

Waxy maize, which is a special strain of maize and yields a starch which is almost completely free from amylose ($< 2\%$)[23], could, because of film-making difficulties, be compared only at the lower thickness, and, as we have seen, irregular relationships between dye concentration and exhaustion allow only restricted conclusions to be drawn. The properties of the pastes from the two varieties are markedly different, dispersion being more ready and complete with waxy maize, and skinning on the surface almost entirely absent.

The general indications (Table III) are that small increases in the equilibrium exhaustion from starch occur in passing from potato to normal maize and from normal to waxy maize.

Schoch[17] had earlier given the proportion of amylose in both potato and maize starch as 22%. More recent results of Higginbotham and Morrison[24] are potato 22·1%, maize 28·5%, while Bourne and Peat[23] suggest that waxy maize contains less than 2%. Ignoring the other, not inconsiderable differences in the pastes of these starches, this relates higher exhaustion with increasing amylose content for the first pair, which is not unreasonable if it is considered that the amylose may be present in the films in a more completely crystalline state and is therefore specifically less accessible to dye molecules than the ramified amylopectin. However, the results with waxy maize contradict this conclusion.

There is no doubt, however, of the effects produced by hydrolysis of the starch. From Table III, it is seen that, at two different dye concentrations, exhaustion from acid-hydrolysed maize is markedly higher than from the unhydrolysed starch. Precisely how this hydrolysis may modify the association of dye molecules with the thickener it is difficult as yet to see. Probably the question will remain unanswered until the effect has been related in detail to the action of the acid in modifying the grosser heterogeneities of the pastes. The results with maize starch partly hydrolysed during cooking by the presence of dilute acid are very different from those obtained with a torrefied starch, since films of British gum have very poor exhaustions.

EFFECT OF FILM THICKNESS

In Fig. 2 and 3 are given the results of dye transfer experiments with normal maize starch films of two thicknesses, one approximately half that of the other. The dye content per disc of unit area has been maintained approximately constant, so that initial dye concentration in the thinner film is approximately twice that in the thicker. In spite of this, the equilibrium exhaustion value to cellulose from the thinner film is very little greater. This again calls attention to the complex relation between exhaustion and initial external concentration already commented upon, and shows that the relationships which hold for normal dyeing conditions from an aqueous bath are greatly

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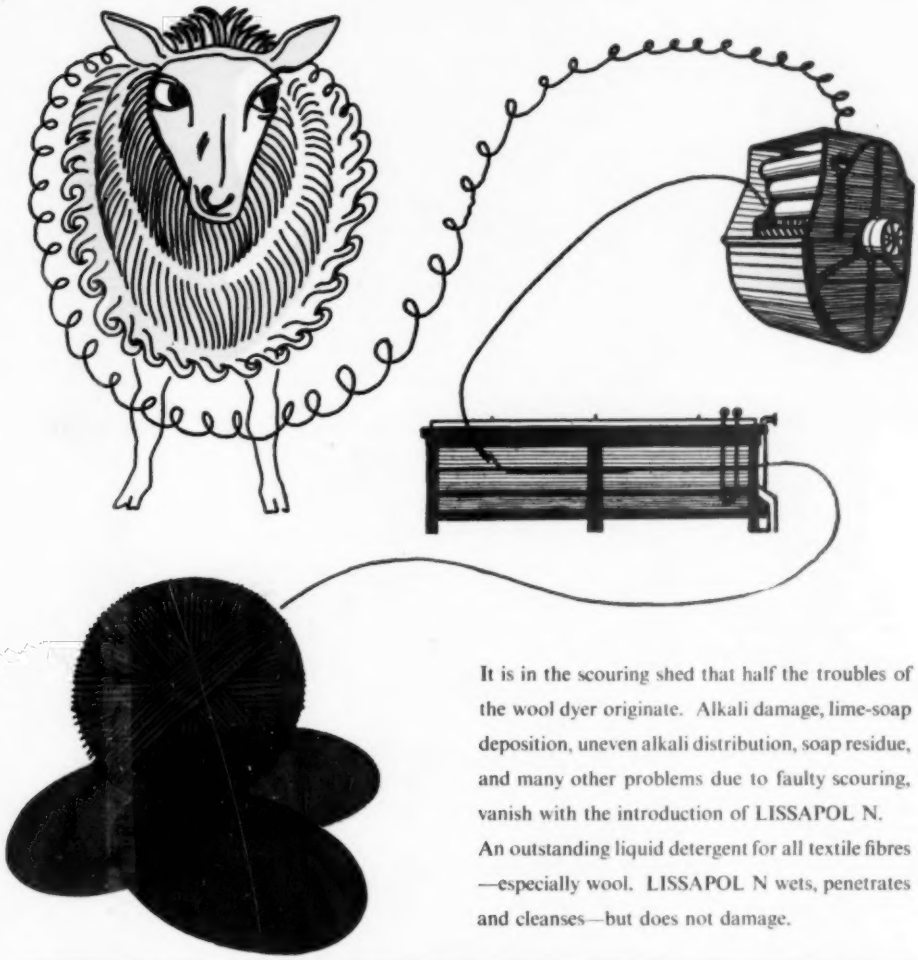

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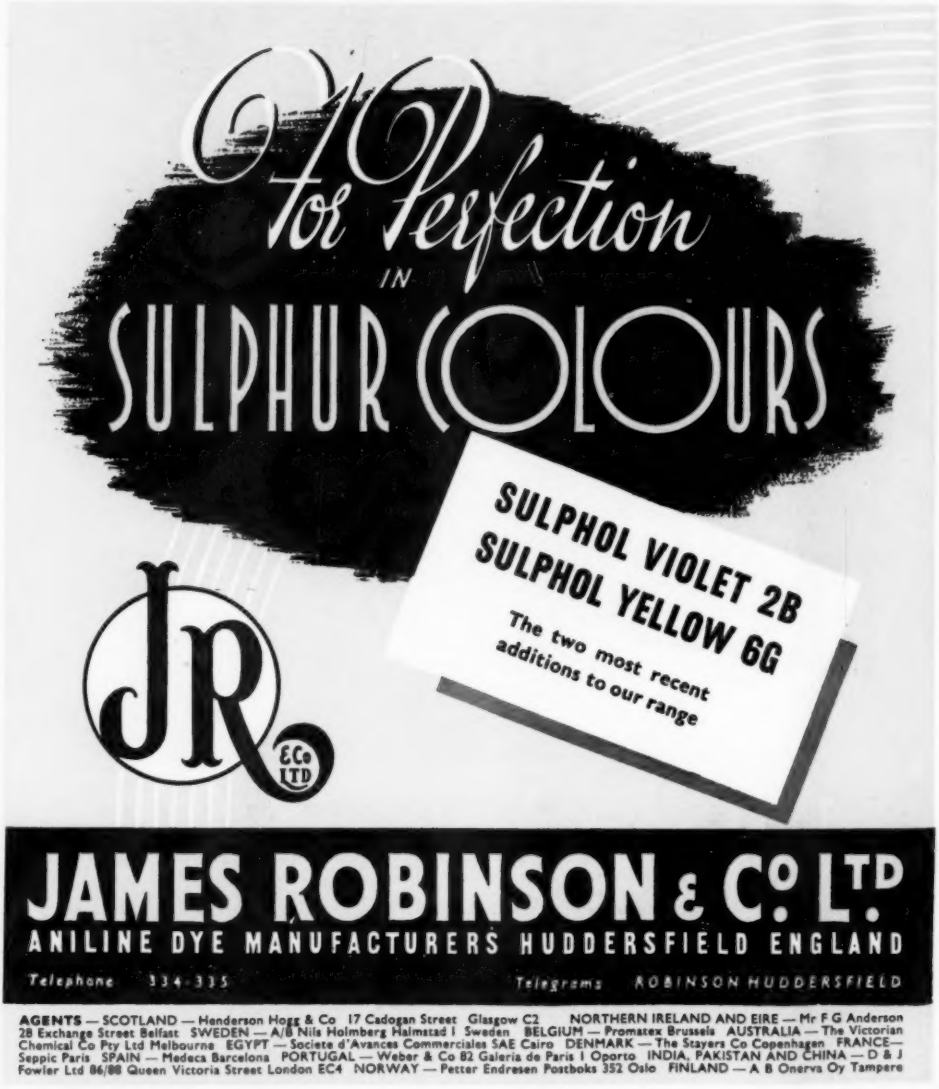
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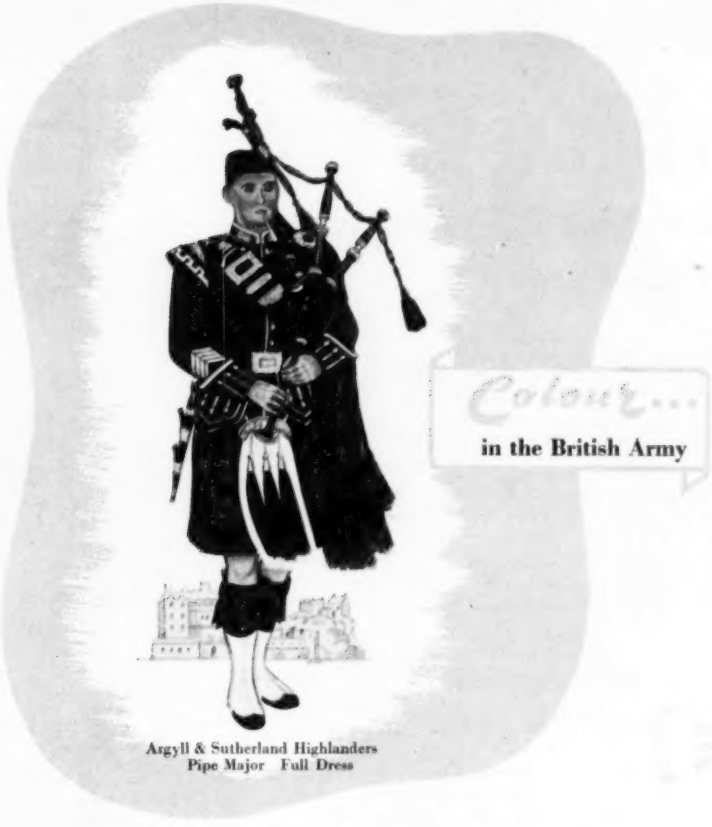

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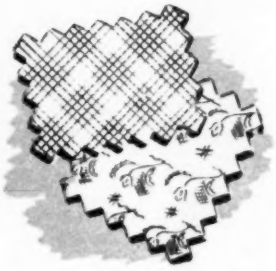

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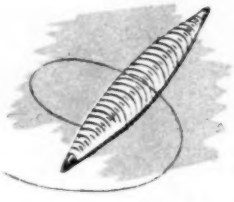
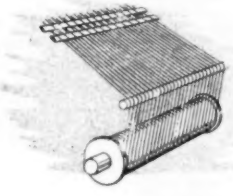


Syton is an anti-slip agent for increasing fibre drag. Syton is recommended for use in woollen, worsted and cotton spinning. It imparts increased strength to the yarn resulting in fewer "ends down" and, by permitting reduction in twist, gives increased production.

Syton does not affect dyeing or finishing and scours out easily.

SANTOBRITE



Santobrite (sodium pentachlorophenate) is a preservative for preventing mildew and similar growths on sized yarns or finished fabrics. Especially suitable for cotton and linen. It enables larger batches of size to be mixed and kept, and allows sized yarns to be stored damp and pliable without risk of mildew or discoloration. Applied in the final wet finishing operation, it preserves fabrics in transit and in store.

The names 'Santomerse', 'Syton', and 'Santobrite' are registered trade marks.

MONSANTO CHEMICALS LIMITED, Victoria Station House, London, S.W.1

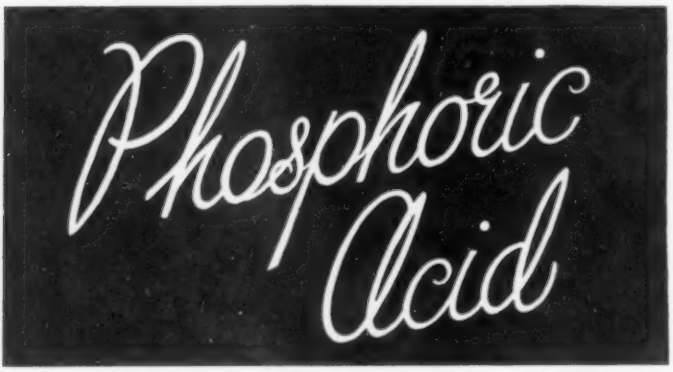
ALCOPOL O

DI-OCTYL ESTER OF SULPHO-SUCCINIC ACID

UNIVERSALLY ACKNOWLEDGED
THE MOST EFFICIENT WETTING
PRODUCT

ALLIED COLLOIDS (MANUFACTURING) CO LTD
BRADFORD MANCHESTER LONDON

For the DYEING OF WOOL in all stages



has the undermentioned advantages over other acids—

1. Less damage to wool on prolonged boiling or in acid milling, compared with Sulphuric Acid.
2. Better and faster shades, due to elimination of iron reaction.
3. Saving in time and more level results in the dyeing of weakly acid and chrome colours.
4. More even dyeing of imperfectly scoured material, due to buffering action, and consequent stabilisation of pH.

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is at your disposal

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BRAMLEY LEEDS

modified when dyeing takes place from a film of thickener.

We wish to acknowledge our indebtedness to the City of Manchester Education Committee for a scholarship which assisted one of us (D. A. R.) to take part in this work.

Department of Textile Chemistry
College of Technology
The University
Manchester

Received 24th October 1950)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Dr. Williams and his Green Dye for Cotton

The Editor

Sir,

In his letter of 5th July 1950, Mr. H. Hampson[1] asked about Dr. R. Williams and his green dye for cotton. This is presumably the Dr. Williams referred to on p. 346 of James Napier's *Manual of the Art of Dyeing* (Glasgow 1853), where it is stated that a Dr. Williams in 1773 was awarded £2000 by Parliament for discovering a fast green and yellow dye upon cotton. This dye consisted of weld and a secret mordant, supposed to be either tin or tin and bismuth. The colour was not fast, but Dr. Williams "caused his specimens of dyed yarn to be woven into pocket handkerchiefs, and gave them to be worn in the pockets of those who were afterwards to attest to the goodness of his dye, and as handkerchiefs worn in the pockets were not exposed to the action of the sun and air, this want of permanence was not discovered until some time after the reward had been paid for an invention which proved of little or no value."

Yours truly

C. O. Clark

The Priory
Bolton
Bradford
Yorks.

13th December 1950

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Notes

Proceedings of the Council

At a meeting of the Council, held at the offices of the Society, 32–34 Piccadilly, Bradford, on 10th January 1951, the proceedings included the following items of interest—

International Congress of Textile Scientific Research— It was reported that an invitation had been received from the Institut Textile de France to present a paper at this congress, to be held in Lille on 8–9th May 1951. In reply, it had been suggested that Dr. R. H. Peters would give a paper on the *Measurement and Practical Significance of the Redox Potentials of Vat Dyes*.

Bye-laws Committee— It was resolved to set up a small committee to make suggestions for the co-ordination of the Bye-laws of the Society, removing anomalies and thus rendering the Bye-laws mutually consistent. In addition to the *ex-officio* officers of the Council, Messrs. G. B. Angus, H. Foster, and F. L. Goodall were invited to serve on the committee.

Report of the Society's Medals Committee— It was resolved, after consideration of the Committee's report, to award the Perkin Medal to Professor J. B. Speakman, Gold Medals to Dr. P. W. Cunliffe and Mr. H. Foster, and a Bar to the Silver Medal to Mr. L. A. Lantz.

Annual Dinner 1951— It was reported that Dr. C. J. T. Cronshaw had accepted the Society's invitation to be the chief guest at the Annual Dinner to be held on 30th March 1951.

Nomination of President-elect— Mr. H. H. Bowen was nominated President-elect.

Membership — Nineteen applications for ordinary membership and four for junior membership were approved.

Death of Members

We regret to report the loss by death of Messrs. J. T. Hodgson, E. F. James, J. Pilkington, and Frank J. Smith.

Meetings of Council and Committees February

Council— 14th
Publications— 20th
Colour Index Editorial Panel— 5th
Diploma— 9th
Moth and Dermestid Beetle Proofing— 27th
Bye-laws— 14th

The Society of Dyers and Colourists of Australia

Report for the Session 1949-50

In presenting this, the second Annual Report under the 1948 Constitution, the Council is very pleased to report that what they anticipated last year has come to fruition, namely the formation of a Section in South Australia. This is now properly constituted, and has proved a very live and enthusiastic addition to our Society. The year's activities are summarised as follows—

COUNCIL— This has met twice, and on the second occasion had the pleasure of welcoming Mr. Wainwright, the Chairman of the South Australian Section, to its deliberations. At the first meeting for the year it confirmed a further term to Mr. L. P. Rendell as President. The Council has closely investigated the possibility of an Australian-wide Conference, but expense and accommodation problems made this plan impracticable in the period under review, though all members are well cognisant of the fact that this is a most desirable aim and will continue to study it. It was found that certain portions of the Constitution were not quite clear, and accordingly certain propositions to clear this up will be placed before the Annual General Meeting for ratification. All members present at the Council meetings are well satisfied that this body is performing very valuable work in developing a national consciousness in the Society.

MEMBERSHIP— A sharp rise in membership has taken place, mainly on account of the formation of the South Australian Section, which led to a net gain of 29 members. The figures are now— 3 life, 353 other, a gain of 50 for the year, which is a healthy sign.

MONTHLY MEETINGS— The Sectional Committees have been most active in placing before their members a good selection of speakers and a wide variety of subjects. The actual programmes are shown in the attached appendixes and reflect great credit on the organisation. We want again to thank the many public-spirited gentlemen who have come forward and made these talks possible.

SOCIAL— This side of our activities has been well catered for, all sections holding annual dinners, whilst Victoria and New South Wales both had successful balls. The Victorian Section inaugurated a new idea during the year, when in November their monthly meeting was held in Ballarat; this enabled many members and their wives to spend an enjoyable week-end in the country and to meet a number of Country members. In this regard our thanks go to the Directors of Messrs. E. Lucas & Co., who made their factory available for inspection and loaned their canteen as our social centre for the week-end. This proved such a successful venture that preliminary arrangements are already in hand to visit another Provincial City in the same way.

The Victorian Section played its portion of the President's Golf Trophy competition, but results of the other Sections are not yet to hand, so no decision on the winner is yet available.

LIBRARIES— These continue to expand, and the policy of bringing new additions to the monthly meetings has proved an idea to get greater circulation; in this regard thanks go to our Honorary Librarians, who are keen and efficient in this trust. Our thanks in Victoria are extended to Mrs. Mardel, who kindly donated a copy of *Fibre Science*.

SECTIONAL COMMITTEES— The foregoing reflects the very hard and conscientious job they have done, and our thanks are due to the members who have served on them.

PUBLICATION— The Council through their Publication Subcommittee has kept this side of our activities going along steadily, and we continue to get much valuable assistance and co-operation from Mrs. Mardel, the Editress of *The Textile Journal of Australia*. Our thanks are due for the way our case is presented every month. We have not yet been able to attain our ambition of having plenty of matter from our members and have to thank the Society of Dyers and Colourists for the right to reproduce articles from their *Journal*. Our thanks go to all the authors who have allowed their papers to be published, also to a number of contributors.

PUBLICITY— The Council has taken the opportunity to place details of our activities before a Committee of the C.S.I.R.O., which is organising a Directory of Scientific and Technical Societies.

HISTORY— It is pleasing to report that the Victorian Section have a history nearly ready for publication, whilst steps are being taken to prepare the same data in New South Wales.

SUMMARY— This, we feel, will give members a feeling of achievement and will be a spur to continue the expansion of the activities of the Society. In conclusion, as the inaugural Honorary General Secretary and on the verge of my retirement, I feel I must pay a tribute to the President, Mr. L. P. Rendell. A great deal of the success so far gained can be laid at his door. He has at all times kept before us that this is an *Australian* Society, and only things which are good for the whole of the Society are good enough for him. I feel you will join with me in expressing our thanks to him for what he has done and will continue to do in our interests.

F. D. STEVENS
Honorary General Secretary

APPENDIX I

PAPERS PRESENTED TO VICTORIA SECTION

Fibre to Stocking— A Dyer's Review of the Hosiery Industry	R. Mayston
Application of Synthetic Resins to Wool	M. Lipson
Modern Trends in the Dyeing of Wool	H. Crook
Metomega Colours (film)	Commentary by W. J. Waldie
Sir William Henry Perkin, his Life and Work	N. Lawson
Preparation of Coloured Leather	F. Parkin
Screen Printing Technique	R. Waterworth

Personnel Practice in relation to Dyehouse Administration R. W. R. Glasgow

APPENDIX II

PAPERS PRESENTED TO NEW SOUTH WALES SECTION

Mercerisation of Cotton and Aniline Black J. Chadwick
Metachrome Dyeing with Metomega Chrome Colours (film) Talk by H. H. Buckley
Colorimeters C. H. Giles (read by F. Eastwood)
Bleaching of Cotton Fabrics E. Brecher
New Developments in Dye Vat Controllers F. Blezzard
Textile Plastics F. Curtis

APPENDIX III

PAPERS PRESENTED TO SOUTH AUSTRALIAN SECTION

Technical Aspects of pH W. Russell
Water and its Industrial Applications A. K. Beenie
Modern Trends in the Use of Detergents P. B. Law
Processes relating to the Wool Sheepskin Industry W. J. Waldie
Synthetic Detergents in Wet Processing Textiles G. R. James
Cotton Textiles W. H. Wainwright
The Use of Chemicals in the Dyehouse E. I. Noble

New Books and Publications

The Physical Chemistry of Dyeing

By T. Vickerstaff. Pp. viii + 416. London: Oliver & Boyd (published for Imperial Chemical Industries Ltd.) 1950. Price, 42s. 0d.

During the last half-century there has been steady progress in the bringing of hitherto obscure phenomena within the framework of physical chemistry. The process of dyeing was at one time regarded as essentially a "colloidal" phenomenon, obeying only its own special rules. Vickerstaff's book shows that a clearer and more useful picture is obtained if dyeing is regarded in the light of classical physical chemistry, the thermodynamics of Willard Gibbs being particularly valuable. The book is a notable achievement and represents a landmark in the application of pure science to industrial problems.

The author has eschewed a historical approach, but has attempted to give a contemporary review of modern dyeing theory. He has presented the various alternative viewpoints with a good deal of fairness and impartiality, so that the reader unfamiliar with the subject may find himself a little confused. In a branch of applied science undergoing rapid growth, such a treatment is preferable to a didactic standpoint. One may, perhaps, regret a little the lack of some historical account of the process whereby, during the last twenty years, the subject has been brought within the compass of physical chemistry, the historical approach being very often the easiest as well as the most illuminating.

The use of thermodynamics is exhaustive, and on the whole lucid, though there is some slight carelessness in that strict use of symbols which is so essential if confusion is to be avoided. The most serious criticism of this section, however, is a certain failure always to emphasise sufficiently clearly the distinction between the dye molecule and the coloured ion, and this is the crux of the whole modern theory of dyeing with direct and acid dyes. The choice between the thermodynamic system of Willard Gibbs and that of van't Hoff is a smaller matter, though the general use of the former is more in line with practice. It is a little surprising to note that the earlier treatments based on the Donnan equilibrium fail to be recognised as "thermodynamic".

The reader may be struck by the paucity of experimental data on which the theoretical treatment rests, these being confined mainly to quantitative measurements of dye absorption under various conditions of dye and salt concentrations and of temperature. There is no exact knowledge of activity coefficients, of the distribution of the added salt, or of the electrical potentials which play such an important rôle. These are matters for further research; the author has made a very workmanlike job of his review of the subject written in 1946. His book should form an indispensable part of the equipment of every student of textile chemistry, and it may surprise many a "pure" scientist to see that science and technology have, in this field, become so closely linked.

S. M. NEALE

Proceedings of Heating and Drying Conference

Institute of Textile Technology. Pp. 88. Charlottesville, Va. 15th April 1948.

This booklet gives a summary of the proceedings of the conference, and has two main points of interest. One is that it gives an impression not only of what was known but also, and equally important, of what was not known in this field in the United States. The second point of interest is constituted by the new data presented here on the thermal and monetary efficiencies of dryers of different types. A survey was made of hot-air and can-type dryers, and comparisons were made of these traditional types with more modern ones using "infra-red" and "high-frequency" as the means of energy transfer to the material to be dried.

Data are given about the requirements in pounds of steam or, depending on the type of heating, on the number of kilowatt-hours required to evaporate a pound of water from textile materials. Data are also given on the costs of steam and electricity, so that the relative costs of the different processes were ascertained. In addition there are diagrams and photographs of drying equipment, but there is no bibliography or index.

This publication is welcome as presenting some new facts in a field which is remarkable for its barrenness of hard facts. This is surprising when the great magnitude of textile drying operations is considered, for it is estimated here that, in slasher

drying in the United States alone, nearly a million tons of water is evaporated each year.

J. M. PRESTON

Technique of Organic Chemistry

Volume I

Physical Methods of Organic Chemistry

Parts I and II

Edited by A. Weissberger. Second revised edition 1949. Pp. xii + 1072 (Part I) and xi + 1024 (Part II). New York and London: Interscience Publishers. Price, cloth, $12.50 or £5 each part.

This excellent collection of monographs, the first edition of which has been reviewed previously (J.S.D.C., **62**, 219 (1946)— Part I; **63**, 57 (1947)— Part II), has now been augmented to the extent of over 50% by five new chapters and other additions and revisions. The chapters in Part I are—

I— *Temperature Measurement* J. M. Sturtevant
II— *Temperature Control* J. M. Sturtevant
III— *Determination of Melting and Freezing Temperatures* E. L. Skau and H. Wakeham
IV— *Determination of Boiling and Condensation Temperatures* W. Swietoslawski and J. R. Anderson
V— *Determination of Vapour Pressure* G. W. Thomson
VI— *Determination of Density* N. Bauer
VII— *Determination of Solubility* R. D. and M. J. Vold
VIII— *Determination of Viscosity* T. E. McGoury and H. Mark
IX— *Determination of Surface and Interfacial Tension* W. D. Harkins
Parachor G. W. Thomson
X— *Determination of Properties of Monolayers and Duplex Films* W. D. Harkins
XI— *Determination of Osmotic Pressure* R. H. Wagner
XII— *Determination of Diffusivity* A. L. Geddes
XIII— *Determinations with the Ultracentrifuge* J. B. Nichols and E. D. Bailey
XIV— *Calorimetry* J. M. Sturtevant
XV— *Microscopy* E. E. Jelley
XVI— *Determination of Crystal Form* M. A. Peacock
XVII— *Crystallochemical Analysis* J. D. H. Donnay

Part II contains—

XVIII— *X-Ray Diffraction* I. Fankuchen
XIX— *Electron Diffraction* L. O. Brockway
XX— *Refractometry* N. Bauer and K. Fajans
XXI— *Spectroscopy and Spectrophotometry* W. West
XXII— *Colorimetry, Photometric Analysis, Fluorimetry, and Turbidimetry* W. West
XXIII— *Polarimetry* W. Heller
XXIV— *Determination of Dipole Moments* C. P. Smyth
XXV— *Conductometry* T. Shedlovsky
XXVI— *Electrophoresis* D. H. Moore
XXVII— *Potentiometry* L. Michaelis
XXVIII— *Polarography* O. H. Müller
XXIX— *Determination of Magnetic Susceptibility* L. Michaelis
XXX— *Determination of Radioactivity* W. F. Bale and J. F. Bonner, Jr.
XXXI— *Mass Spectrometry* D. W. Stewart

Of the entirely new chapters, (I) covers its subject matter fully, and describes the useful galvanometer–photocell negative-feedback amplifiers; (II) deals with both continuous and on–off types of temperature controllers, including an original electronic thyratron relay for resistance-thermometer control; (V) deals fully with ordinary and ultra-low ($< 1\mu$.) pressure ranges, includes micro and semi-micro techniques, and contains useful sections on fitting curves to vapour pressure–temperature data, estimating vapour pressures from meagre data, and calculating heats of vaporisation. (XIII) includes the treatment of sedimentation velocity and equilibrium, and gives illustrative applications to determining molecular weights and shape of molecules of dyes, proteins, sera, high polymers, etc.; (XXVI) is mainly concerned with the Tiselius moving-boundary method of measuring electrophoretic mobility, and contains a short section on applications to separating the constituents of complex high-molecular mixtures. Electrophoresis, which in the first edition accounted for merely an unsatisfactorily short section in the chapter on Conductometry, is now justifiably raised to full chapter status, in view of its ever increasing importance in practical and theoretical studies of large molecules in pure and applied chemistry.

Some chapters have new sections added. Thus, (XV) has a short section on electron microscopy, (XXII) is augmented by sections on nephelometry, and light-scattering and its application to molecular weights and particle shape of high polymers. (XXI) now contains sections on light sources, valuable in connection with polarimetry, refractivity, photometry, and photochemical reactions, as well as with spectroscopy and spectrophotometry.

Some deficiencies noted in the first edition have been repaired. A full subject index accompanies each part. Chapter VIII now includes a treatment of the Couette type of viscometer; (IX) contains a full treatment of the pendant drop method for surface tension; (X) includes the two Harkins–Jura and the B.E.T. methods for determining the surface area of a solid; (XIV) deals with various determinations of importance in organic chemistry; and (XV) treats phase contrast, ultra-violet, fluorescence, and dark-field (ultra-microscopic) microscopy.

Production, type, and binding remain excellent. One or two errors in page number references due to revision were noted (e.g. on p. 1779, references to pp. 1102 and 1103), and also a misprint on p. 1783, last paragraph (pH 4 as compared with pH 5 in Fig. 24). The unfortunately high price will doubtless severely restrict the sale of these books in this country, but they can nevertheless be strongly recommended to all concerned in stocking reference libraries.

H. I. STONEHILL

Reports on German Industry

I.G. Farbenindustrie A.-G. Höchst a. M.
Flow Sheets and Diagrams relating to Production Processes used in the Various Plants at Höchst

FD 1145/50* (PB 55,997, 73,842, 55,996, 70,204, 55,947; Microfilms AA 115, D 23, D 24, CC 132, BB 109).

Five microfilms (1791 frames) of flow sheets and diagrams of the production processes used in the Höchst plants for dyes, intermediates, auxiliary agents, and other chemicals. The diagrams usually give full details of the construction of the plant including materials used. An index is included. C. O. C.

I.G. Hauptlaboratorium
Chemical Index

FDX 885* (O.T.S. Microfilm M 112–140).

Microfilm of approximately 30,000 cards in German giving data relating to chemical products under trade names marketed by European, American, and a few Japanese firms. Each card gives the chemical name of the product or its analysis together with notes on application, constitution, patents, and literature references. Dyes form the largest single section, but a wide variety of products for all sorts of uses are included. C. O. C.

I.G. Hauptlaboratorium
Chemical Index

FDX 892*—Addendum to FDX 885 (O.T.S. Microfilm M 112–140).

A selection of some of the German trade names, excluding dyes and pharmaceuticals, contained in the index. The name, maker, and constitution or description are given in each case. C. O. C.

Anorgana G.m.b.H., Gendorf
Mepasins, Kogasins, and Hostapons

FD 1167/50* (PB 70,344).

A collection of 18 papers, written in German in 1941–43, dealing with the preparation of aliphatic sulphonic acids and their uses in preparing detergents and auxiliary products. C. O. C.

Anorgana G.m.b.H., Gendorf
Ramasits and Other Waterproofing Agents

FD 1171/50* (PB 7034).

Microfilm copy of 9 papers written in German—

(1) *Works Process for Producing the Water-repellent Agent Hö 1/67* [*Stearyloxybetaine*] (G. Wagner, 21st Dec. 1939)— Detailed instructions are given for making stearyloxybetaine, a good water-repellent agent, and the necessary octadecyl chloromethyl ether. Replacement of dimethylamine by diethylamine yielded a product of almost the same properties as the original (cf. (4) below).

(2) *The Problem of a Wash-resistant Water-repellent Finish* (G. Wagner, 27th Dec. 1939). Results of trials on the chemical modification of mercerised cotton.

(3) *Influence of Stability and Charge on an Aqueous Suspension of a Water-repellent Agent* (Seydal, 15th February 1940) — Trouble is sometimes caused in the use of aqueous suspensions of water-repellent agents because the emulsifying agents used compete with the water-repellent agent in affinity for the fibre. *Persistol WS*, hydroxymethylstearamide, can be made into stable suspensions having any desired charge by using a neutral soap as the emulsifying agent.

(4) *Continuous Production of Alkoxymethylbetaines of High Molecular Weight (Hö 1/92)* (H. Wagner, 15th March 1941)— Stearyl chloromethyl ether and diethylaminoacetic ester are heated at 58°c. until reaction is complete. The product is immediately saponified with aqueous caustic soda to yield the betaine. The water-repellent obtained by use of diethylamine (Hö 1/92) differs from that where dimethylamine is used (Hö 1/67) in that the "ripening" temperature is 15–20°c. lower.

(5) *Water-repellent Finish fast to Washing and Milling* (V. Fink, 27th November 1941)— Addition of Palmin P to Persistol VS and Hö 1/107 improves their water-repellent effect.

(6) *Various Methods of Evaluating Water-repellency* (Dietrich, 13th February 1943)— The following tests are described and criticised— (*a*) water head method, (*b*) artificial rain method, (*c*) water drop method, and (*d*) suction method. Methods (*c*) and (*d*) are the simplest and most accurate. Work is also described on the way various water-repellent agents are selectively absorbed by the two components in wool–viscose rayon mixtures.

(7) *Improvement of Ramasits I, WD conc., and K conc.* (Wappes, 4th May 1936)— Ramasit I was rendered stable to mechanical agitation by using bone glue as a stabiliser. Addition of formic acid to Ramasit K conc. brought it up to the efficiency of Imprägnol. Ramasit WD was improved by using resin soap instead of stearin soap.

(8) *Improvement of the Ramasits* (Wappes, 10th October 1936)— Ramasit K improved by addition of formic acid tended to foam and so to give spotty finishes. This was prevented by addition of 0·2–0·5% Lorol extra (C_{12}–C_{14} fraction of coconut or palm kernel alcohol). Addition of 0·25% Emulphor A gives even better results. Ramasit KW was rendered extremely stable by just mixing the water-insoluble components and then emulsifying them in the aqueous phase at 70°c.

(9) *Attempts to Improve Ramasit K conc.* (Wappes, 18th December 1936)— The stability of the emulsion was greatly improved by replacing the paraffin wax by one of the I.G.'s synthetic ceresins. Until sufficient supplies of this were available, Ramasit K conc. was still made from paraffin wax emulsified with 5% of resin acid at pH 6 in absence of Nekal, the emulsion being then mixed with aqueous aluminium formate in the usual manner. C. O. C.

I.G. Farbenindustrie A.-G., Hoechst
Colloid Mills— Reflex Blue AG— Intermediates
NN'-Ethyleneureas as Leather and Textile Auxiliary Agents

FD 1157/50* (PB 70,248).

Microfilm copy of seven papers written in 1939—

(1) *Comparing the Grinding Properties of Several Colloid Mills*

(2) *Attempts to reduce the Cost of making Reflex Blue AG*

(3) *Reaction Products of ββ'-Dihydroxy- and ββ'-Dichloro-diethyl Disulphide*

(4) *Derivatives of Ethyleneimine*

(5) *Production of Intermediates 1934–38*— An account of the work done by the various Hoechst departments making intermediates, including production figures, past and future plans, etc.

(6) *NN'-Ethyleneureas— Properties and Use as* [*Leather and*] *Textile Auxiliaries*— These products can be used for animalising cellulosic fibres and rendering PC and glass fibres capable of being dyed. One of the products can be used instead of formaldeyde for hardening coating colours for leather. Five such ureas having good water-repellent properties are cited, while others are softening agents for cellulosic fibres.

(7) *Keten and Diketen* [*in Plasticiser, Resin, and Dye Manufacture*]. C. O. C.

I.G. Farbenindustrie A.-G., Leverkusen
Dyes, Eulans, Synthetic Tannins, Applications of Diisocyanates

FDX 891* (PB 82,171; Microfilm T 29).

Routine progress, quarterly, and annual reports covering 1936–43 from various research laboratories. They are

* Material available for inspection at, and photocopies available from, T.I.D.U., Board of Trade, Lacon House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

mainly concerned with work on a large variety of dyes, including improvements in the manufacturing processes and plants, and mothproofing agents. C. O. C.

I.G. Farbenindustrie A.-G., Ludwigshafen
6th and 7th AKo (Azo Dyes Commission) Meetings December 1933 and July 1934

FD 1173/50* (PB 17,677).

A film of 233 research reports (1101 frames) from Ludwigshafen, Leverkusen, Mainkur, and Höchst on the preparation of azo dyes, examination of competitive products, etc. C. O. C.

I.G. Farbenindustrie A.-G., Hoechst
Non-coloured Self-coupling Dyes—Organic Chemicals—Intermediates—Sirius Supra Blue F3GL

FD 1158/50* (PB 70,248).

Microfilm of 18 papers written in German in 1938. They include the following two—

(1) *Non-coloured Self-coupling Dyes [for Application to Mixtures of Wool and Viscose Rayon]*
(2) *Manufacture of Sirius Supra Blue F3GL.*

C. O. C.

I.G. Farbenindustrie A.-G., Hoechst a. M.
Alizarin and Vat Dyes

FD 1152/50* (PB 70,248).

Microfilm copy of 16 papers read at I.G. technical meetings in 1938–1940—

(1) *1-Methylbenzanthrone Derivatives and Dyes* (Schneider)
(2) *Dioxazine Dyes containing Carboxyl Groups* (Langbein and Thiele)
(3) *Blue Acetate Rayon Dye from Tetrahydroquinizarin* (Koch)
(4) *1-Hydroxy-4-bromoanthraquanine-2-ω-methanesulphonic Acid and Dyes therefrom* (Schlichemeier and Meisaner)
(5) *Halogenated Dibenzpyrenequinones [Preparation, Properties, and Use as Intermediates]*
(6) *Dyes from Halogenated Dibenzpyrenequinones* (Corell)
(7) *Alkoxyperylenetetracarboxylic Acids and Dyes from them*
(8) *Hydroxytrimellitic Acids and Dyes from them* (Eckert and Schilling)
(9) *Yellow Chrome Dyes of the Chrome Fast Yellow 5 GD Type* (Eckert)
(10) *Derivatives of 1-Methylbenzanthrone* (Schneider and Runne)
(11) *Verification of* B.P. *Application 14331* [? 1939] *of Ciba* (Besler)
(12) *1:5:9:10-Anthracenetetracarboxylic Acid and Dyes from it* (Becker)
(13) *Indigoid Dyes containing Trifluoromethyl Groups*
(14) *Water-soluble Carbonamide [Anthraquinone and Indigoid Pyridinium Chloride] Dyes and their Use* (Corell)
(15) *Chrome Dyes of the Chrome Fast Yellow 5 GD Type* (Dieferbach)
(16) *Anthrachrysonedialdehyde* (Roemer). C. O. C.

I.G. Farbenindustrie A.-G., Hoechst
Rhodulin Heliotrope 3B

FD 1154/50* (PB 70,248).

A microfilm of 8 reports, one of which, written in 1938, states that the residues of the pyrolusite used in preparing this dye could be freed from the dye much better if processed in acetic acid solution. Application of this observation had led to economies in steam and time, increase of 78% in the yield, and reduction by 20% of the aniline needed, and had halved the cost price. C. O. C.

I.G. Farbenindustrie A.-G., Hoechst
Vat and Acid Dyes

FD 1150/50* (PB 70,248).

Microfilm of the following nine papers written in German in 1938–40—

(1) *Anthraquinone Vat Dyes containing Trifluoromethyl Groups—IV [Derivatives of 5-Amino-4'-ω-trifluoromethylanthraquinone-2:1(N)-1':2'(N)-benzacridone]*
(2) *Anthraquinone Vat Dyes containing Trifluoromethyl Groups—V*
(3) *Work on Vat Blue P113 [a Monochloro Derivative of 2-Phenylbenzanthrone-pyrazoleanthrone]*
(4) *New Anthraquinone Acid Dyes fast to Washing and Milling*
(5) *2-(Chlorophenyl)-quinizarins and Acid Dyes derived from them*
(6) *Indigo Vats from Indoxyl Crystals and Indigo Paste*
(7) *Navy Blue Vat Dye [derived from 4-Aminotrifluoromethylacridone]*
(8) *Vat Dyes from Substituted* peri-*Indandiones*
(9) *Vat Dyes for Wool [Helindon Yellow CG, pp'-Dichloro-2:5-phenylamino-1:4-benzoquinone, and other Helindon Dyes]* C. O. C.

I.G. Farbenindustrie A.-G., Hoechst
Dyes and Intermediates

FD 1151/50* (PB 70,248).

Microfilm of the following eight papers written in German in 1938–40—

(1) *Substituted 1-Aminoanthraquinones as Bases for Naphtol AS Dyes* (in two separate parts)
(2) *[Anthralan Dyes of the] Bromamine Series*
(3) *Preparation of* p-*Substituted Isatinanilides [as Raw Material for Indanthren Printing Black B]*
(4) *Indigosol Yellows and Oranges from Azo Dyes*
(5) *Preparing a Stable Powder of Indigosol Blue IBC*
(6) *Some Azo Compounds of the Anthraquinone Series*
(7) *Indanthren Bordeaux RR [and Orange GR]*
(8) *Anthraquinones containing Trifluoromethyl Groups*

C. O. C.

I.G. Farbenindustrie A.-G., Ludwigshafen
Application of Lucrylan II Solutions

FD 2619/48* (PB 17,252).

A photocopy of working instructions in German, dated 18th July 1940, for the application of various solutions of Lucrylan II, both pigmented and uncoloured, for coating fabrics for ladies' raincoats, oiled cotton fabric, bedding materials, and weather-resistant batiste. C. O. C.

* Material available for inspection at, and photo copies available from, T.I.D.U., Board of Trade, Lacon House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

Abstracts from British and Foreign Journals and Patents

(The Titles of Patents are abridged and modified)

I—PLANT; MACHINERY; BUILDINGS

Philblack Carbon Black Plant. W. G. Norris. *International Chem. Eng.*, 31, 543–545 (Dec. 1950).

An account illustrated by photographs and a flow sheet of the plant of Philblack Ltd. at Avonmouth, designed to produce 50,000,000 lb. of furnace black, Philblack A and O, per annum. C. O. C.

PATENTS

Continuous Liquid Treatment of Freshly Spun Rayon Threads. Oscar Kohorn & Co. Ltd. and J. L. Costa. *U.S.P.* 2,520,594.

Web Tension Control. Cameron Machine Co., J. S. Scheuermann, and T. N. Carter. *U.S.P.* 2,521,413.

Where a carriage, capable of to-and-fro movement, supports a tension-control roll that engages a bight or loop in a running web, the use of weights for obtaining a constant pull on the carriage is avoided by operating and controlling the carriage by fluid pressure, preferably air pressure. C. O. C.

Hosiery-drying Apparatus. Paramount Textile Machinery Co. and H. Richter. *U.S.P.* 2,519,981.

A number of internally heated forms are so mounted that

their positions may be changed by the operator leaning against a movable bar, thus leaving his hands free at all times to manipulate the hosiery on the forms. C. O. C.

Setting of Nylon Stockings. Paramount Textile Machinery Co. and H. Richter. *U.S.P.* 2,520,062.

Drum Washing Machines. Achille Serre Ltd. and A. C. Parsons. *B.P.* 647,439.

By supplying the washing liquid to the interior of the drum and allowing it to drain away from the drum through an aperture therein, there is little or no filtering effect during washing. In addition, the washing capacity of the liquor is used to the full, more clothes can be washed in a drum of given size as compared with hitherto known machines, and the washing time is considerably reduced and is independent of the speed of rotation of the drum. C. O. C.

Hardening Felt Hoods. Merrimac Hat Corpn. *B.P.* 646,718.

Dyeing of Fur Skins. P. Poser. *B.P.* 647,101.

The skins are packed into a closable container, each skin having its flesh side touching the flesh side of another skin or being folded with its flesh side on the inside. The fur side of each skin touches the fur side of another skin. The skins are packed under such pressure that when liquid is passed through the container there is less resistance to its flow between the fur sides of the skins than between the flesh sides. C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Production of Sodium Chlorite—Survey of Prevalent Manufacturing Methods. G. Holst. *Ind. Eng. Chem.*, **42**, 2359–2371 (Nov. 1950).

A detailed electrochemical and thermodynamical analysis of the various reactions involving the reduction of chlorine dioxide to produce sodium chlorite has led to the choice of lead oxide (litharge) as the most suitable reducing agent available. An experimental study of the lead oxide process has shown that excellent yields of sodium chlorite are obtained together with a valuable by-product, lead dioxide. The favourable economics of the process should be of importance in future technical developments. W. K. R.

PATENTS

Water-soluble Cellulose Compound. Courtaulds Ltd., F. Ward, and E. E. Tallis. *B.P.* 647,321.

Cellulosic material is impregnated with an aqueous solution of sulphuric acid and cyanamide, and is then baked. The conditions are such that the final water-soluble product contains not $< 25\%$ by weight of combined sulphuric acid. The resulting material has fire-resistant properties. It can be used as a carrier or core yarn in the production of effect threads or fabrics, or it may be used as a size which is readily removed by washing. C. O. C.

Carboxyalkyl Ethers of Carbohydrate Gums. General Mills Inc. and O. A. Moe. *U.S.P.* 2,520,161.

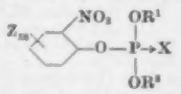
Carboxyalkyl ethers of carbohydrate gums and especially their alkali-metal salts are readily dispersible to form much clearer sols than do the untreated gums. The carboxyalkyl ethers of gums of galactomannan and glucomannan types form gels from aqueous sols on addition of ferrous sulphate, aluminium sulphate or chloride, etc. These ethers are useful as thickening, sizing, and finishing agents, protective colloids, and stabilising agents for emulsions. C. O. C.

Emulsifying Agents to produce Water-in-oil Emulsions. F. A. Möller. *B.P.* 647,133.

A mixture of compounds capable of reacting to yield a product containing the residues of a polyhydric alcohol, of a higher fatty acid, and of a polybasic carboxylic acid is heated to cause them to react. The compounds and their relative proportions are so chosen that the product is soluble in oil but not in water and is capable of producing by the "steam emulsion" test an emulsion of 20 c.c. of water in 20 c.c. of a 0·25% solution of the product in neutral freshly refined monkey-nut oil, from which emulsion not > 5 c.c. of oil separates at 93–95°c. in the test. The "emulsion test" is as follows—20 c.c. of the oil to be tested is placed in a graduated glass test tube 20 cm. deep, 2·3 cm. in diameter, and with walls 0·8–1·4 mm. thick. The oil is put in it at room temperature, and the tube placed in a water-bath at 19–26°c. Steam is then passed into the oil so as to maintain its temperature at 88–91°c. until the total volume of liquid in the tube is 40 ± 3 c.c. Admission of steam is stopped as soon as this volume is reached, and the tube immediately transferred to a water-bath kept at 93–95°c. The volume of the separated oil layer is measured to the nearest c.c. every 30 sec. C. O. C.

o-Nitrophenyl Dialkyl Phosphates and Thiophosphates. American Cyanamid Co. and J. H. Fletcher. *U.S.P.* 2,520,393.

Compounds of formula—



(R^1 and R^2 = Alk, aralkyl, or Ar; X = S or O; Z = Hal or Alk; m = 1 or 2) are fungicides and insecticides; e.g. they may be used for controlling the black carpet beetle. C. O. C.

Alkyldimethylbenzylammonium Naphthenates. Nuodex Products Co. Inc. and M. Nowak. *U.S.P.* 2,519,924.

Compounds of formula $NR^1R^2R^3R^4\}X$ (X = radical of a water-insol. oil-sol. acid; R^1, R^2, R^3, and R^4 = same or dissimilar, subst. or unsubst. organic radicals), e.g. dodecyldimethylbenzylammonium naphthenate, are excellent mildew-, rot-, and insect-proofing agents. C. O. C.

Siliceous Dispersions. Silicon (Organic) Developments Ltd., C. Shaw, and W. E. Langrish-Smith. *B.P.*647,528.

Stable dispersions are obtained by adding to a lower alkyl orthosilicate water in excess of that required for complete hydrolysis and in presence of a water-miscible solvent for the orthosilicate. They may be used as anti-slip agents for textiles, imparting anti-tack properties to uncured natural rubber, and other purposes for which surface roughness is necessary. C. O. C.

Alkoxy-, Aryloxy-, or Aralkoxysilanes—Fireproofing and Delustring Agents. Silicon (Organic) Developments Ltd., D. A. W. Hill, C. Shaw, and W. E. Langrish-Smith. *B.P.* 647,537.

A mixture of a tetra-alkyl and/or tetra-aryl and/or tetra-aralkyl orthosilicate or their incompletely hydrolysed and intermolecular condensates (the degree of hydrolysis and intercondensation corresponding to that obtained by adding $< 1{\cdot}5$ mol. of water per mol. of orthosilicate or when this water is present in the alcohol or phenol from which the silicon ester is made), when mixed with not $> 5\%$ of its weight of an inorganic metallic amide or hydrazine, can be diluted with water. Such compositions are useful as fireproofing and delustring agents. C. O. C.

Acceleration Vulcanising Agents suitable for use in presence of Furnace Blacks. Monsanto Chemicals Ltd. and W. M. Morgan. *B.P.* 647,652.

A mixture of *cyclo*hexylbenzthiazylsulphenamide and 2:4-dinitrophenyl benzthiazyl sulphide leads to satisfactory vulcanisation in presence of furnace black, especially finely divided furnace black. C. O. C.

Emulsion Polymerisation of Ethylene. L. Seed and I.C.I. Ltd. (XIII, p. 129.)

Carbon Black as Catalyst for Curing Alkyd Resin Adhesive Compositions. Johnson & Johnson (Gt. Britain) Ltd. (XIII, p. 129.)

Polysiloxane Resins. British Thomson-Houston Co. Ltd. (XIII, p. 129.)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Investigation of Substantive Furoyl Dyes. I—Acylacetarylides. R. Andrisano. *Boll. Sci. Facoltà Chim. ind. Bologna*, **8**, 4–6 (1950): *Chem. Abs.*, **44**, 9675 (25th Oct. 1950).

The preparation and m.p. of several acylacetarylides are

given, where acyl includes acetyl, furoyl, 5-methyl-2-furoyl, and benzoyl. The method consists in boiling the ethyl acylacetate with 1 mol. of a primary arylamine (e.g. aniline, *o*- or *p*-anisidine) for a few minutes. The *a*-furoylacetoarylides can be cyclised to the corresponding 4-furyl-2-hydroxyquinolines.

III— Monoazo Derivatives of Acylacetanilides. *ibid.*, **8**, 10–11 (1950): *Chem. Abs.*, **44**, 9675 (25th Oct. 1950).

Azo derivatives are obtained by coupling diazotised amines (aniline, *o*- and *p*-toluidine, etc.) with acylacetanilides, where acyl = acetyl, furoyl, and benzoyl. The products were crystallised from acetic acid, and the crystalline form, colour, and m.p. were recorded. E. S.

Synthesis of Chlorantine Fast Red 5BL and 7BL, and Chlorantine Fast Bordeaux BLL. T. Nishi and U. Miyashita. *J. Soc. Chem. Ind. Japan*, **45**, Suppl. binding 304–305 (1942) [in German]: *Chem. Abs.*, **44**, 10,323 (10th Nov. 1950).

The structure of some Chlorantine Fast dyes (J.S.D.C., **57**, 125 (1941)) was confirmed by synthesis. Thus, diazotised 4-aminoazobenzene-4′-sulphonic acid was coupled under alkaline conditions with benzoyl-J acid to give Chlorantine Fast Red 5BL, with *m*-nitrobenzoyl-J acid to give Chlorantine Fast Red 7BL, and with *m*-aminobenzoyl-J acid to give Chlorantine Fast Bordeaux BLL. E. S.

Azomethin Dyes from 4-Anisylidene- and 4:4′-Anisylidenebis-pyrazolones. G. W. Sawdey, M. K. Ruoff, and P. W. Vittum. *J. Amer. Chem. Soc.*, **72**, 4947–4949 (Nov. 1950).

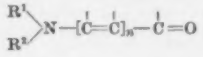
4-Anisylidene- and 4:4′-anisylidenebis-pyrazolones undergo oxidative condensation with *NN*-disubstituted *p*-phenylenediamines to produce magenta azomethin dyes. These reactions involve replacement of the anisylidene group by a *p*-phenylenediamine residue. C. O. C.

Fading of Methylene Blue by X-Rays. Ya. L. Shekhtman, A. A. Krasnovsky, and I. V. Bereshchinsky. *Doklady Akad. Nauk S.S.S.R.*, **74**, 767–769 (1st Oct. 1950).

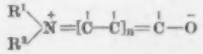
The extinction coefficients, for light of wavelength 660 mμ., of solutions of Methylene Blue in dil. H_2SO_4 were determined before and after exposing the solutions to X-rays. For a given soln. the value of the extinction coefficient after exposure fell with increase of dosage in an almost linear fashion. Addition to the soln. of quinone, quinol, glucose, or glycerol led to a considerable reduction in fading. The results are in accord with the theory that radiochemical reactions of this type depend essentially not on the direct action of the radiation on the solute, but on its action on water, forming active radicals and ions, which then react with the solute. The molecules of the added organic substances are able to compete successfully with the dye molecule for reaction with the active species, and hence have a blocking action. A. E. S.

Structure of *mero*Cyanine Dyes. I. I. Levkoev, N. N. Sveshnikov, and E. B. Lifshits. *Doklady Akad. Nauk S.S.S.R.*, **74**, 275–278 (11th Sept. 1950).

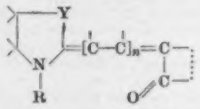
The work of Kiprianov and his co-workers on dyes of the general formula—



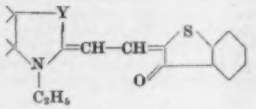
or, in its mesomeric form—



(R^1 and R^2 = hydrocarbon residues; n = 0, 1, 2, or 3) has shown that they may be divided into two groups, according to the way in which the position of the absorption maximum varies with the nature of the solvent. For a member of the first group, e.g. an indoaniline, the absorption maximum is displaced towards the short waves when the solvent is replaced by one of lower dielectric constant; for members of the second group the reverse is the case. It is supposed that in dyes of the first group the purely covalent mesomer makes a preponderating contribution to the actual structure, and the use of a solvent of lower dielectric constant leads to a further displacement towards the purely covalent form, leading to a hypsochromic effect, for it is the structure in which the two mesomers make equal contributions which shows the maximum depth of colour. Dyes of the second group approximate to the zwitterion in structure, and the increase in the contribution of the covalent form, which occurs on passing to a solvent of lower dielectric constant, now leads to a bathochromic effect. The typical *mero*cyanines—



(Y = O, S, Se, CR_2, >C=C<, NR, or others; R = hydrocarbon residue; n = 0, 1, 2, or 3) have been hitherto considered to belong to the first of the above groups, i.e. to approximate in structure to the purely covalent form. An examination is now made of seven *mero*cyanines of the general formula—



the nature of the basic heterocyclic residue being varied. In a series of solvents of diminishing dielectric constant, ranging from methanol to *n*-hexane, the wavelength at the absorption maximum either fell progressively (for residues of low basicity) or rose at first and then fell (for residues of higher basicity). It is concluded that, in solution, a *mero*cyanine may have a structure approaching either extreme, depending on the basicity of the nitrogen-containing residue. The wavelength at the absorption maximum is tabulated for each *mero*cyanine and for the two corresponding dyes of symmetrical structure (a carbocyanine and a thionaphthenomonomethinoxanine). The value for the *mero*cyanine is always less than the mean of the values for the corresponding symmetrical dyes by an amount which, when the solvent is chloroform, falls in a fairly regular manner as the basicity of the nitrogen-containing residue increases, but, when the solvent is the more polar ethanol, shows a rather irregular fall to a minimum followed by a rise. An attempt is made to interpret these facts in accordance with the suggested mesomeric behaviour of the dyes. A. E. S.

Quenching of the Fluorescence of Chlorophyll and of Magnesium Phthalocyanine and the Reaction between these Pigments and Quenching Substances. V. B. Evstigneev, V. A. Gavrilova, and A. A. Krasnovsky. *Doklady Akad. Nauk S.S.S.R.*, **74**, 315–318 (11th Sept. 1950).

Measurements were made of the intensity of the fluorescence of solutions of chlorophyll and of magnesium phthalocyanine in toluene, ethanol, and pyridine, and the effect of additions of various substances was determined. It was found, in agreement with previous work in methanol and ethanol, that the most effective quenchers are oxidising agents. The photochemical reactions, if any, between these added substances and the pigments were also examined; there was no direct relation between the quenching effect of the added substance and its ability to react photochemically. It was concluded that the photochemical reactivity of the pigment is associated not with the excited electronic state of 10^{-8}–10^{-7} sec. duration, but with the long-lived diradical (triplet) state. A. E. S.

Comparison of the Photochemical Properties of Chlorophyll, Phæophytin, Phthalocyanine, and Magnesium Phthalocyanine. V. B. Evstigneev and V. A. Gavrilova. *Doklady Akad. Nauk S.S.S.R.*, **74**, 781–783 (1st Oct. 1950).

Experiments are described which show that the presence of magnesium in the molecules of chlorophyll and magnesium phthalocyanine increases their tendency to undergo photochemical oxidation in visible radiation, as compared with the corresponding magnesium-free substances—phæophytin and phthalocyanine. The reverse is the case for photochemical reduction. The reactions were carried out in soln. in toluene or pyridine. Dissolved

oxygen served as oxidising agent; ascorbic acid and phenylhydrazine hydrochloride were used as reducing agents. A. E. S.

PATENTS

Stabilising Phenylenediamines and Aminophenols with Amine–Sulphur Dioxide Reaction Products. Universal Oil Products Co. and R. B. Thompson. *U.S.P.* 2,521,425.

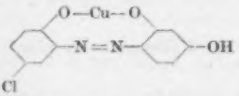
Addition of a little of the reaction product of sulphur dioxide and an organic amine stabilises phenylenediamines and aminophenols against discoloration on being stored for a long time. C. O. C.

Yellow, Copperable, Substantive Disazo Dyes. J. R. Geigy A.-G. *B.P.* 647,103.

Yellow, copperable, substantive dyes are made by coupling 1 mol. of an *NN'*-diacetoacetylbenzidine (which may have the usual substituents in the 3:3' positions) with 1 mol. of a diazotised *o*-aminophenol and 1 mol. of a diazo compound with an *ortho* substituent capable of giving metallic complexes (OH, COOH, O·Alk, etc.). Alkoxy groups in the 3:3' positions of the benzidine give good fastness to washing, and the most valuable dyes are those in which the first diazo component is 6-benzoylamino-2-aminophenol-4-sulphonic acid and the second diazo component is an unsulphonated *o*-aminophenol or anthranilic acid, especially 5-acetylamino- and 5-benzoylamino-anthranilic acid. Thus *NN'*-diacetoacetyldianisidine is coupled first with 1 mol. of diazotised 6-benzoylamino-2-aminophenol-4-sulphonic acid, then with 1 mol. of diazotised 5-acetylaminoanthranilic acid. The product gives reddish yellows on cotton when applied by the aftercoppering method. E. S.

Azoic Browns using Metallised Resorcinol–Azo Compounds as Coupling Components. General Aniline & Film Corpn. and C. Streck. *U.S.P.* 2,515,743.

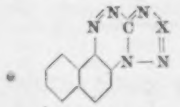
The metallised *o*-carboxy- and *o*-hydroxyarylazo-resorcinols described in *U.S.P.* 2,135,433 are useful coupling components in the production of azoic browns by continuous dyeing and printing processes, because of their low substantivity towards cellulose fibres. The dyeings produced by development with a diazonium compound vary in hue throughout the material, but uniformity is secured by aftertreatment in an acid solution of sufficient strength to remove alkali metal bound by the dye on the fibre. Thus cotton cloth is impregnated continuously with an alkaline solution of—



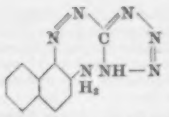
(made by coppering 2-amino-4-chlorophenol→resorcinol), and then passed through a solution containing the zinc chloride double salt of 5-chloro-2-methoxybenzenediazonium chloride. The material is then passed through aqueous hydrochloric acid of 10° Bé. After rinsing and soaping a uniform yellow–brown dyeing is obtained. E. S.

***N*-Cyclic Compounds from Azo Dyes — Intermediates and Dyes.** General Aniline & Film Corpn., H. W. Grimmel, and J. F. Morgan. *U.S.P.* 2,515,728.

Naphtho-[2:1-e]-tetrazolo-[5:1-c]-*as*-triazines and naphtho-[2:1-e]-*s*-triazolo-[5:1-c]-*as*-triazines—



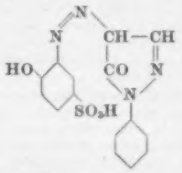
(X = N or subst. C) are made by coupling diazotised 5-aminotetrazole or 5-amino-1:2:4-triazoles with a β-naphthylamine capable of coupling in the α-position *ortho* to the amino group, and effecting ring closure of the resulting azo compound by elimination of the β-amino group. Thus diazotised 5-aminotetrazole is coupled with β-naphthylamine to give the red azo compound—



On heating in dil. H_2SO_4, NH_3 is eliminated to give naphtho-[2:1-e]-tetrazolo-[5:1-c]-*as*-triazine (X = N). E. S.

Azo Derivatives of 1-Aryl-5-Pyrazolones. American Cyanamid Co. and R. D. Kleene. *U.S.P.* 2,512,251.

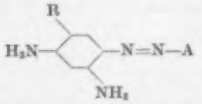
Azo derivatives of 1-aryl-5-pyrazolones unsubstituted in the 3-position are made by coupling diazo compounds with arylhydrazones of formylacetic ester, when cyclisation occurs simultaneously. Depending on the diazo compound used and on the arylhydrazone, the products are azoic pigments, water-soluble dyes (which may be substantive), or lakes. Thus by coupling diazotised *o*-aminophenol-*p*-sulphonic acid with ethyl formylacetate phenylhydrazone under alkaline conditions, the monoazo pyrazolone compound—



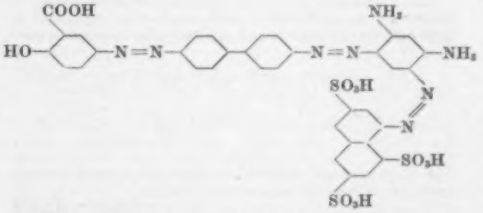
is formed. By metallising with chromium acetate, an orange wool dye is produced. E. S.

Brown Trisazo Leather Dyes. J. R. Geigy A.-G. *B.P.* 646,753.

Brown trisazo dyes for leather are made by coupling 1 mol. of tetrazotised benzidine with 1 mol. of an *o*-hydroxycarboxylic acid of the benzene series and 1 mol. of a sulphonated monoazo compound of the chrysoidine series of formula—



(R = H or CH_3; A = the radical of an aniline or naphthylamine-polysulphonic acid). The resulting dyes, containing > 1 sulpho group, are more soluble in acid media than the similar direct cotton browns with only one sulpho group and hence give dyeings on leather of better penetration. An example is—



made by coupling 1 mol. of tetrazotised benzidine with 1 mol. of salicylic acid and 1 mol. of the the chrysoidine-type dye 1-naphthylamine-3:6:8-trisulphonic acid→*m*-phenylenediamine. E. S.

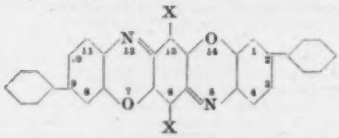
1:4-Diacylaminoanthraquinone Derivatives — Vat Dyes. Ciba Ltd. *B.P.* 646,171.

Modification of *B.P.* 605,466 (cf. J.S.D.C., **65**, 34 (Jan. 1949)). Pink vat dyes of the 1:4-diacylaminoanthraquinone type have as one acyl group the residue of a pyridine- or quinoline-carboxylic acid, and as the other the residue of an *NN*-dialkylsulphamylbenzoic acid or the

corresponding *NN*-dialkylene compound in which the alkylene part forms a ring, e.g. morpholine. Thus 1-amino-4-(*p*-*NN*-dimethylsulphamyl)benzoylaminoanthraquinone is added to a mixture of pyridine-β-carboxylic acid and thionyl chloride in nitrobenzene at 120–130°c., and stirred for 2 hr. R. K. F.

Triphendioxazine Derivatives—Pigments. I.C.I. Ltd. and R. W. G. Preston. *B.P.* 646,099.

Violet pigments comprising 2:9-diphenyltriphendioxazines, of formula—



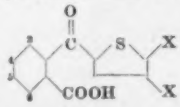
(X = H, Cl, or Br) are prepared by heating 2:5-bis-4′-diphenylylamino-1:4-benzoquinone (or the corresponding 3:6-dichloro or 3:6-dibromo derivative) in presence of an acid chloride and an oxidising agent, or a substance, e.g. *m*-nitrobenzenesulphonyl chloride, combining both functions. Thus 2:5-bis-4′-diphenylylamino-3:6-dichloro-1:4-benzoquinone, made by refluxing 4-aminodiphenyl with chloranil and anhyd. sodium acetate in ethanol, is heated at 250°c. for 2·5 hr. in chloronaphthalene with *m*-nitrobenzenesulphonyl chloride. R. K. F.

Sulphuric Esters of Leuco Vat Dyes. General Aniline & Film Corpn. and J. Taras. *U.S.P.* 2,507,944.

Sulphuric esters of leuco vat dyes are made by treating an aqueous alkaline solution of the leuco vat dye with the sulphur trioxide addition compound of pyridine or α-picoline. Thus, all parts being by weight, Jade Green (15) is dissolved in KOH (37·5) and sodium hydrosulphite (17·5) in water (500). While at 35–40°c. under nitrogen, the addition product (50) prepared by treating pyridine (500) in chloroform (1500) with SO_3 (521) at 10–20°c., and distilling off the chloroform, is introduced. R. K. F.

2-Thienoyl-*o*-benzoic Acids and Derived Thiophanthraquinones. du Pont, H. R. Lee, and V. Weinmayr. *U.S.P.* 2,513,570.

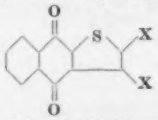
Methyl-2-thienoyl-*o*-benzoic acids of formula—



(one X = CH_3, the other X = H) are prepared by condensing α- or β-methylthiophen (1 mol.) with phthalic anhydride (1 mol.) in presence of aluminium chloride (2·2 mol.) in nitrobenzene at 50–85°c.

U.S.P. 2,513,571.

The compounds of the preceding patent are cyclised by treatment at 110–130°c. with aluminium chloride in nitrobenzene to give the methylthiophanthraquinones—



The cyclisation may follow the initial condensation without isolation of the thienoyl-*o*-benzoic acid.

U.S.P. 2,513,572.

Halogen-substituted 2-thienoyl-*o*-benzoic acids carrying one or more Cl or Br atoms in positions 3, 4, 5, and 6 are made by condensing halogenated phthalic anhydrides with the Grignard reagent of an α-halogenothiophen. Thus magnesium turnings and a crystal of iodine are added to dry ether, and an ethereal soln. of α-bromothiophen is introduced. The whole is refluxed under nitrogen, and added after cooling to a soln. of 3:6-dichlorophthalic anhydride in benzene. After refluxing again, water is added, and the whole boiled for 1 hr. The product is separated by adding MgO, removing the benzene–ether layer, and acidifying the remainder.

U.S.P. 2,513,574.

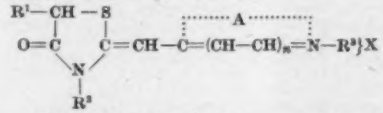
The compounds of the preceding abstract are cyclised to the corresponding thiophanthraquinones by treating with sulphuric acid at 170–175°c., with aluminium chloride in nitrobenzene at 125–130°c., or with phosphorus pentoxide in nitrobenzene at 130–135°c.

U.S.P. 2,513,573.

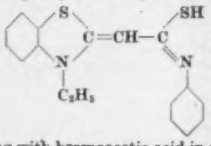
Nitro-2-thienoyl-*o*-benzoic acids with the NO_2 group in position 3, 4, 5, or 6 are prepared from the corresponding nitrophthalic anhydride by the method of *U.S.P.* 2,513,572 (above). R. K. F.

Thiazolone Cyanine Dyes—Photographic Sensitisers. General Aniline & Film Corpn., L. C. Hensley, and T. R. Thompson. *B.P.* 646,900.

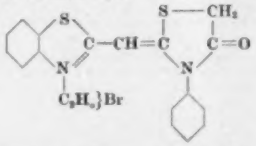
Thiazolone cyanine dyes of formula—



(A = residue of a heterocyclic nitrogenous nucleus of the type common in cyanine dyes with 5 or 6 atoms in the heterocyclic ring, e.g. oxazole, thiazole, etc.; *n* = 0 or 1; R^1 = H, CH_3, or Hal; R^2 = alkyl, aryl, aralkyl, or allyl; R^3 = alkyl or aralkyl; X = an acid radical) are made by heating substituted thioamides with a halogeno- or dihalogeno-acetic acid or with an α-halogenopropionic acid. Thus 2-methylbenzothiazole is *N*-ethylated and then heated with phenyl *iso*thiocyanate to yield—



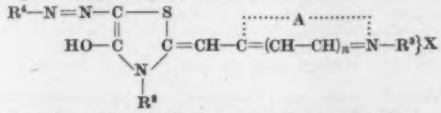
which on heating with bromoacetic acid in *n*-butanol gives the thiazolone cyanine dye—



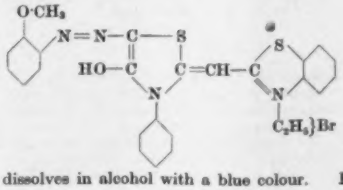
E. S.

Azo Compounds of Thiazolone Cyanine Dyes. General Aniline & Film Corpn. and T. R. Thompson. *B.P.* 646,888.

Many of the thiazolone cyanine dyes described above combine in substance or on the fibre with diazo compounds to give azo dyes of general formula—



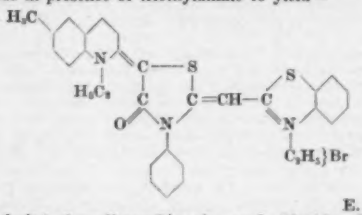
(R^4 = the residue of a diazo compound, aromatic or heterocyclic, with or without sulpho and other substituent groups; R^2, R^3, A, *n*, and X have the meanings given for *B.P.*646,900 (above)). Thus, diazotised *o*-anisidine is coupled with the 3-ethyl-3′-phenyl-4′-hydroxythiazolothiacyanine bromide described above, to give the azo compound—



which dissolves in alcohol with a blue colour. E. S.

Trinuclear Thiazolone Cyanine Dyes. General Aniline & Film Corpn. and T. R. Thompson. *B.P.* 646,896.

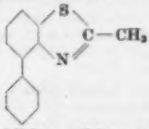
Trinuclear cyanine dyes are made by treating the thiazolone cyanine dyes described in *B.P.* 646,900 (above) with a quaternary *cyclo*ammonium salt of the type commonly used in cyanine dye syntheses. Thus 2-methylthio-6-methylquinoline ethiodide is boiled in methanol with 3-ethyl-3′-phenyl-4′-hydroxythiazolothiacyanine bromide in presence of triethylamine to yield—



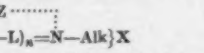
E. S.

2-Methyl-4-phenylbenzthiazole and Methin Dyes therefrom. Kodak Ltd., L. G. S. Brooker, and G. Van-Zandt. *B.P.* 647,542.

2-Methyl-4-phenylbenzthiazole—



is obtained by oxidising *o*-thioacetamidodiphenyl with an alkali-metal ferricyanide. It can be condensed with quaternary *cyclo*ammonium salts of formula—



(n = 0 or 1; L = methin or an alkyl-substituted methin group; Z = atoms to complete a 5- or 6-membered heterocyclic nucleus; X = an acid radical; Y = Cl, Br, I, a thioether group, Ar·NR·[L:L]$_d$–, or Alk·S·L:L–; d = 1–3; R = H, Alk, or acyl). C. O. C.

Colouring Material for Lubricating Oils. Sinclair Refining Co., E. P. Zehner, and O. D. Hunn. *U.S.P.* 2,519,868.

A greenish bloom or fluorescence is given to lubricating oils by adding 0·25–1·0% by weight of the product obtained by distilling a heavy gas oil of boiling range 500–800°F. (obtained in the Thermofor Catalyst Cracking process) down to 10% of its original volume and then blowing the residue with air. C. O. C.

Purifying Rutin. U.S. Secretary of Agriculture, J. F. Couch, J. Naghski, and W. L. Porter. *U.S.P.* 2,520,127.

Rutin is separated from impurities, particularly quercetin, by dissolving the mixture in an anhydrous lower aliphatic alcohol and, if necessary, immediately filtering to remove any material insoluble in alcohol. Upon standing the rutin alcoholate precipitates and is recovered by filtration. It is then hydrolysed with water, and the rutin filtered off and dried at 110°c. C. O. C.

Seed for Production of Titanium Dioxide. M. J. Mayer. *U.S.P.* 2,519,389.

Philblack Carbon Black Plant. W. G. Norris. (I, p. 120.)

Carbon Black as Catalyst for Curing Alkyd Resin Adhesive Compositions. Johnson & Johnson (Gt. Britain) Ltd. (XIII, p. 129.)

VI—FIBRES; YARNS; FABRICS

Existence of a Subcuticle or Intermediate Membrane in Wool and Hair. E. H. Mercer; H. Zahn. *Melliand Textilber.*, 31, 694–695 (Oct. 1950).

The thin tubes isolated from wool fibres by Lehmann, Elöd, and Zahn were considered by them to be an intermediate membrane situated beneath the cuticle. It has now been demonstrated by E. H. Mercer, however, that these tubes are a layer from the outside of the cuticle. Wool fibres were coated with gold metal and then treated with phenol and digested with trypsin; the tubes isolated in this way were found to be still coated with the metal.

In reply to this paper H. Zahn describes the isolation of tubes by first treating wool with formamide and then digesting with pancreatin. Microscopic observations during the course of the digestion show that these tubes undoubtedly lie beneath the scales. Similar tubes have been isolated from human hair by the action of caustic soda.

Mercer reviews the situation by stating that he has established the location of the tubes which were isolated in the manner first described by Lehmann *et al.* and erroneously described as a subcuticle. The tubes which Zahn has latterly investigated are of different origin and may justifiably be termed an intermediate membrane. F. A.

Changes produced in Wool by Processing. S. Blackburn. *Chem. and Ind.*, 718–723 (4th Nov. 1950).

The composition of wool and the importance of cystine and cysteine determinations are discussed. The methods of examining changes in wool discussed are— partition chromatography for estimating changes in the amino acid composition, staining techniques, which generally assess relative damage and uniformity of treatment, and physicochemical methods such as examining changes in elasticity and strength. The changes produced in wool, in scouring, carbonising, bleaching, setting, and shrink-resisting, are discussed from the point of view of its structure and the chemical changes produced in the amino acid constitution. The different types of treatment are assessed in terms of the cysteic acid content. W. J. M.

Felting Propensity of Carrotted Hairs. P. Hückel. *Melliand Textilber.*, 31, 689–693 (Oct. 1950).

Facilitation of the felting of rabbit hairs by carrotting has been ascribed to differential hydrolysis, swelling, and supercontraction of the fibres. The process has been further investigated by treating heavily carrotted hairs in baths of various composition. Berg's Curling Test was used for measuring the efficacity of the treatments. Curling was not manifest in boiling methanol, acetone, or benzene, but took place on addition of NaOH to the methanol or of H_2SO_4 to the benzene, so that curling is evidently connected with the polar groups in the fibre. Swelling but not curling was observed in glycerol and propylene glycol. Blocking of acid groups by reaction with chrome alum and of basic groups by reaction with Irgatan B prevented curling. The relation between supercontraction and curling is discussed. The work done in bending a fibre is calculated to be a function of the fourth power of the diameter of the fibre. This explains why a sheep's wool which was coarser than rabbit hair required very severe treatment to cause curling. Supercontraction is thus unlikely to be accompanied by curling when a certain fibre diameter is exceeded. F. A.

Developments in the Production of Synthetic Protein Fibres. H. G. Fröhlich. *Melliand Textilber.*, 31, 663–666 (Oct. 1950).

Summary of recent literature on the production of synthetic fibres from proteins, especially casein, zein, and ground-nut protein. F. A.

Properties of Polyurethan Fibre. E. Elöd and H. G. Fröhlich. *Melliand Textilber.*, 31, 759–762 (Nov. 1950).

The absorption of NaOH and various acids and dyes by polyurethan fibre has been measured. The results are analogous in many ways to those obtained with protein fibres. The absorption of NaOH begins at pH 7–8, increases slightly up to pH 11·5, and then shows a sudden increase, reaching a maximum at pH 13·5–14. Absorption of HCl begins at pH 2 and increases rapidly below pH 1. Weaker acids are absorbed at higher pH values. The absorption of the acid dyes Crystal Ponceau 6R and Eriorubine 2B begins at pH 2 and reaches a value of 1·5% in N-HCl. Absorption of basic dyes begins at pH 7, reaches a maximum at pH 9–10, and then falls rapidly. Steaming of polyurethan fibre causes a decrease in the absorption of basic dyes and an increase in that of acids and acid dyes. It is believed that these results may be explained by hydration of terminal *iso*cyanate groups in the fibre followed by decarboxylation. The hydration produces carboxyl groups, which are responsible for the absorption of

alkali and basic dyes. On steaming, the carboxyl groups are removed, leaving primary amino groups, which give increased absorption of acids and acid dyes. F. A.

PATENTS

Viscose Rayon, Foils, etc. Oscar Kohorn & Co. Ltd. and J. L. Costa. *U.S.P.* 2,521,450.

Means by which the reaction time may be lessened are described. C. O. C.

Viscose Rayon. H. A. Kuljian. *U.S.P.* 2,519,382.

Considerable savings in manufacturing costs and improved quality of filaments are obtained by degasifying the coagulating bath and then saturating it with an inert gas, at the same time blanketing the surface of the bath with an inert gas. This prevents production of free sulphur and its deposition on the filaments and spinnerets. C. O. C.

Lubricant for Viscose Spinning Process. Courtaulds Ltd. and J. Wharton. *B.P.* 646,963.

Formation of rock-like deposits of sulphur on thread-guides, etc. is prevented by adding to the acid coagulating bath a lubricating agent consisting of an aliphatic alcohol or acid having >10 C or a condensate of such an alcohol or acid with ethylene oxide. W. G. C.

Recovery of Salts of Copper and Tartaric Acid from Waste Cuprammonium Cellulose Solutions. American Bemberg Corpn. *B.P.* 647,350.

Fibres from Polymers of Carbamyl- and Sulphamyl-styrene. Eastman Kodak Co., J. B. Dickey, and T. E. Stanin. *U.S.P.* 2,520,917.

A polymer suitable for the production of fibres is obtained by polymerising a mixture containing 9–75% by weight on the polymerisable material of styrene substituted in the ring by a sulphonamide and/or a carboxyamide group and 81–25% of a compound containing a polymerisable $-CH{:}C<$ group, e.g. acrylonitrile. C. O. C.

Composition of Crotyl Cellulose and Compatible Butadiene Copolymer. du Pont and W. H. Charch. *U.S.P.* 2,520,711.

Products suitable for use as fibres, films, coatings, etc. are obtained by cross-vulcanising an unsaturated cellulose derivative, e.g. crotyl cellulose, and a compatible elastomer, e.g. a polymeric diene containing a carbinol group. C. O. C.

Shrink-resistant Fabrics. R. Dogger. *B.P.* 647,223.

The tension imparted to warp threads by winding, etc. with consequent warpwise shrinking in desizing the fabric made from them is eliminated by conducting the individual warp threads directly to the loom from the cops on to which they are spun. If desired, they may be passed through a sizing box on their way to the loom. The cloth formed in the loom is passed into a storing box and not wound on to a beam, thus avoiding stretching the warp during weaving.

B.P. 647,224.

Suitable apparatus is described. C. O. C.

Fabrics formed from Twistless Strands. United States Rubber Co. and B. H. Foster. *U.S.P.* 2,521,055.

A thin, strong, highly flexible, closely constructed fabric, not $>0{\cdot}025$ in. thick, is obtained by using as warp and weft flat strands so that there are about 50 strands to the sq. in., each strand lying as a flat non-twisted ribbon in the fabric. Each strand is compact, strong, flat, several hundredths of an inch wide, and formed of longitudinally parallel fibres permanently bonded together without twist solely by a dried organic adhesive. C. O. C.

Water-soluble Cellulose Compound. Courtaulds Ltd., F. Ward, and E. E. Tallis. (III, p. 121.)

Cross-linked Polymeric Materials. du Pont and P. W. Morgan. (XIII, p. 129.)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Suspending Power of Detergent Solutions. II—Soap–Builder Solutions. A. S. Weatherburn, G. R. F. Rose, and C. H. Bayley. *Canadian J. Res.*, **28 F**, 363–375 (Oct. 1950).

The suspending powers (S.P.) of various alkaline salts, commonly used as soap builders, were assessed by measuring photometrically their efficiency in preventing soil deposition on to cotton cloth shaken in a suspension of carbon black in the builder solution. In the absence of soap, there was little difference in the S.P. of the builders studied, which were generally inferior to distilled water. The addition of builders to soap solutions resulted in a decrease in the S.P. of the soap, this effect increasing with increasing builder concentration. Three types of builder were studied— simple electrolytes, phosphates, and silicates— and within each group the S.P. of a built soap solution increased with decreasing pH. When classified according to their effect on the S.P. of various pure soaps, sodium hexametaphosphate was best, and sodium hydroxide and sodium orthosilicate were worst; there were only small differences between the other builders. This classification, based only on S.P., is not necessarily indicative of the efficiency of builders in the detergent process as a whole. A tentative explanation of the effect of builders on the S.P. of soap solutions is offered, on the basis of interference with the normal adsorption of soap by the carbon particles and, in some cases, the superimposed adsorption of the anion of the builder. W. K. R.

Literature Survey of Textile Bleaching. I— Continuous and Semicontinuous Bleaching— Hypochlorite *versus* Peroxide. A.A.T.C.C. Sub-committee (J. F. Hagen). *Amer. Dyestuff Rep.*, **39**, P 663–P 666 (2nd Oct.), P 703–P 714 (16th Oct.), P 739–P 750 (30th Oct.), P 900 (25th Dec. 1950); **II— Bleaching— General Considerations and Miscellaneous Processes.** *Ibid.*, **39**, P 782–P 786 (13th Nov.), P 820–P 830 (27th Nov. 1950).

Proposed Tentative Test Methods for Evaluation of Continuous Scouring of Raw Grease Wool. A.A.T.C.C. Wool Scouring Committee. (XIV, p. 131.)

VIII— DYEING

Dyeing Properties of Direct Cotton Dyes. E. Köster. *Textil Praxis*, **5**, 657–659 (Oct.), 719–725 (Nov. 1950).

Review of methods for classifying the dyeing properties of direct cotton dyes. The behaviour of the Sirius range of dyes on viscose rayon is summarised in a large table which includes data on the influence of temperature and salt on absorption, amount of dye absorbed in 1 min., and rate of migration. The uses which can be made of such data are discussed. F. A.

Sampling Device for Pressurised Package Dyeing Machines. P. M. Cole and T. W. Stricklin. *Amer. Dyestuff Rep.*, **39**, 801 (27th Nov. 1950).

Yarn dyed in package machines at $>212°$F. may be sampled for matching, etc. without excessive loss of heat by threading the end of yarn from one package through a fine constriction in the wall of the vessel, which is normally sealed. A suitable seal is obtained by the use of a screw clamp on a neoprene tube projecting from a pipe plug installed in the cover of the vessel. Drawings of the device are given. W. K. R.

Relation between Iodine Sorption and Degree of Crystallisation of Cellulose Fibres. II.— Iodine Sorption Method for Examining Dyeing Faults. K. Schwertassek. *Melliand Textilber.*, **31**, 764–769 (Nov. 1950).

An obscure cause of unevenness in dyed materials is the existence of fibres showing differences in physical structure which influence dye absorption. It has been established that the dyeing of cellulose fibres involves the two processes of diffusion and sorption. In order to gain further insight into the relation between structure and dye sorption it is important to separate these two processes. Equilibrium and reversible sorption of iodine with unrestricted diffusion are realised from solutions of iodine in aqueous potassium iodide. In a previous paper (*ibid.*, **31**, 188–192 (March 1950)) it was shown that the iodine sorption measures the internal surface of cellulose fibres and decreases with increase in degree of crystallisation. Measurements of iodine and dye sorption on various fibres show that changes in these properties do not necessarily follow a parallel course. In fact, the manner in which they vary has been found to be an indication of the cause of those dyeing faults which are due to differences in the physical state of fibres. When acetate rayon is saponified, the iodine sorption decreases owing to crystallisation whilst

sorption of direct dyes increases owing to formation of a suitable pore system. Both iodine and dye sorption decrease when hydrate cellulose is subjected to steaming, the decreased iodine sorption being due to increased crystallisation, whilst the decreased dye sorption is due to a coagulation of the amorphous regions of the fibre with reduction in pore size. The changes produced by steaming may occur under less severe conditions; e.g. when viscose is dried, the parts of the material which dry first show no change, but other parts which have been subject to conditions of high humidity and temperature for some time show reduced iodine and dye sorption. Differences in the degree of stretching of fibres do not influence iodine sorption, because the degree of crystallisation remains unchanged, but dye sorption is reduced owing to a change in the pore system. In staple fibre which has been dyed from an alkaline bath any light parts caused by lack of penetration, which have therefore not been subjected to the swelling action of the liquor, have lower iodine sorption than the darker parts. F. A.

PATENTS

Coloration of Cellulose Acetate. T. E. Marchington & Co. Ltd., G. D. Sutton, and O. Boothby. *B.P.* 646,769.

Secondary or acetone-soluble cellulose acetate is coloured with little or no saponification by dyeing with vat dyes in absence of swelling agents but in presence of sodium hydrosulphite stabilised to pH 10·5–11·4 by addition of an alkaline calcium compound, e.g. calcium cyanamide or hydroxide, before it is added to the dyebath. C. O. C.

Metallisation of Non-metallic Surfaces. Standard Telephones & Cables Ltd. and C. F. Drake. *B.P.* 646,921.

Organic polymers having polar groups on the surface, e.g. polymethyl methacrylate, and inorganic materials, e.g. glass or mica, are given a strongly adhering coating of a metal by activating their surfaces, e.g. by treatment with stannous chloride dissolved in hydrochloric acid, and then treating with a metal salt solution in presence of an anionic surface-active agent and a reducing agent. C. O. C.

Properties of Polyurethan Fibre. E. Elöd and H. G. Fröhlich. (VI, p. 125.)

IX—PRINTING

Catalytic Damage in Textile Printing. W. Schönberger. *Melliand Textilber.*, **31**, 636–638 (Sept. 1950).

If regenerated cellulose fabrics printed with alkaline Rongalite pastes are allowed to remain damp either before or after steaming, then chemical degradation is likely to occur in the presence of iron or copper salts. Fluidity values of fabrics printed with a Rongalite paste containing ferrous sulphate and aftertreated with polyamino and polyhydroxy compounds show that these compounds inhibit catalytic degradation. H. K.

PATENTS

Printing on Wool with Phthalocyanine Dyes containing Quaternary or Ternary Salt Groups. I.C.I. Ltd. and R. Thornton. *B.P.* 646,690.

Resist effects are produced on chlorinated wool by printing on a paste containing a reducing agent stable under neutral or acid conditions, e.g. zinc formaldehydesulphoxylate, an inert substance, e.g. China clay, and an acid or salt capable of liberating acid, e.g. citric acid or ammonium thiocyanate, and then padding with a solution of a phthalocyanine dye containing ternary or quaternary salt groups. C. O. C.

Printing of Transparent or Translucent Sheets of Thermoplastic Cellulose Derivatives and Like Polymers. British Celanese Ltd., J. Allan, and H. F. Oxley. *B.P.* 647,105.

Patterns of very good bloom, transparency, and fastness to rubbing are obtained by application of a dye soluble in the material being printed unaccompanied by any substance insoluble in the sheet. The surface to be coloured is simultaneously or subsequently heated to a temperature above the flow-point of the sheet material, and while at that temperature is pressed with a polished metal surface. C. O. C.

Diazotype Composition for Siliceous Surfaces. General Aniline & Film Corpn. and S. C. Slifkin. *B.P.* 647,227.

The diazotype components are mixed into a film-producing solution or plastic dope. So that the film will permanently adhere to liquid-impervious siliceous surfaces, a compatible silicon-containing solvent, e.g. ethyl silicate, or a silicone resin is added to the solution or dope. After coating the siliceous surface the solvents are evaporated by gentle heating; the necessary exposure is then made, followed by development with an alkaline agent, e.g. ammonia gas. The exposed and developed surface is then baked at a moderate temperature to harden the coating. C. O. C.

Colour Correction Mask for Use in Reproducing Multicolour Transparencies. Kodak Ltd., Eastman Kodak Co., D. Dean, and R. C. Houck. *B.P.* 647,168.

Metallisation of Non-metallic Surfaces. Standard Telephones & Cables Ltd. and C. F. Drake. (VIII, this page.)

X—SIZING AND FINISHING

Sizing. J. Bader. *Melliand Textilber.*, **31**, 630–634 (Sept. 1950).

Current practice in warp and hank sizing is outlined, and a comparison of cylinder and air drying of warps shows that both are similar as regards efficiency of drying. It is suggested that an improvement in size film formation might be obtained by the use of infra-red drying. Some of the advantages and disadvantages of linseed oil for hank sizing are also given. H. K.

Practical Experience with the Crease-resistant Finish. W. Gutmann. *Melliand Textilber.*, **31**, 639–643 (Sept. 1950).

The sequence and explanation of operations in the crease-resist finish applied to regenerated cellulose fibres and the commercial importance of the process are described. H. K.

Application of Polyvinyl Chloride to Fabrics. F. Kainer. *Melliand Textilber.*, **31**, 775–777 (Nov. 1950).

A review of the uses of polyvinyl chloride solutions, dispersions, and sheet in textile finishing. F. A.

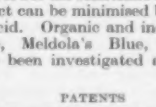
Bacterial Damage to Textiles and its Prevention. M. Nopitsch. *Melliand Textilber.*, **31**, 619–627 (Sept. 1950).

The types of bacteria which attack wool and the conditions favouring their growth are outlined. A method of estimating the degree of degradation microscopically is based upon staining with Cotton Blue in Lactophenol. Chroming, dyeing with chrome dyes, and treatment with Eulans NKF, NK, and N are shown to increase the resistance of wool to bacterial attack. Vat dyeing and treatment with alkalis lower the resistance of the wool to attack, although the effect can be minimised by an aftertreatment with sulphuric acid. Organic and inorganic copper salts, Methylene Blue, Meldola's Blue, and sulphonamide derivatives have been investigated and show some protective action. H. K.

PATENTS

Protecting Cellulose Fibres against Deterioration by Heat Aging. United States Rubber Co., H. M. Buckwalter, and P. D. Montgomery. *U.S.P.* 2,521,446.

Cellulose fibres impregnated with 0·5–7·0% of guanazole—



can be heated for extended periods of time without deterioration. C. O. C.

Boiling or Steaming Bristles, Horsehair, and the like. A. Morer. *B.P.* 646,705.

Curling, unbundling, and rebundling of the bristles are avoided by compressing the imported bundles so that they cannot flex and so that individual bristles cannot move longitudinally by applying clamps around each bundle for its full length together with end covers on each bundle, and

keeping the clamps and end covers on throughout boiling or steaming, drying, and cooling. C. O. C.

Reducing the Extensibility of Nylon Cords. Dunlop Rubber Co. Ltd. and J. W. Illingworth. *B.P.* 647,289.

The extensibility of cold-drawn nylon filaments is reduced by treating them while stretched for 1 hr. with an aqueous fluid at not $<100°$c. The treatment may with advantage be combined with impregnation with a thermosetting resin and curing while the nylon is stretched. C. O. C.

Porous Fibrous Products. A. Talalay. *B.P.* 646,422.

A porous spongelike mass suitable for cushioning material, heat-insulation, etc. is obtained by spraying an aqueous dispersion of an elastomer, e.g. rubber latex, on single or multiple layers of a carded fibre web and then drying. Wool or other fibre having the same dimensional and kink characteristics is employed, and the final product contains a ratio of elastomer to fibre $>1:1$. G. E. K.

Laminated Fabrics. A. Felix, J. J. Horn, M. Horn, and H. Horn. *B.P.* 647,139.

Layers of fabric are bonded together under heat and pressure using as the adhesive a cellulose acetate–solvent-plasticiser mixture. The products are permeable to some extent, and bleeding of dyed material is controlled or eliminated, so that wool or rayon fabrics may be treated. C. O. C.

Cellulosic Materials of Improved Resistance to Chemicals, especially Alkalis. U.S. Secretary of Agriculture and R. E. Reeves. (XI, this page.)

Treating the Surfaces of Porous Webs. Institute of Paper Chemistry, G. D. Knight, and B. W. Rowland. (XI, this page.)

Cross-linked Polymeric Materials. du Pont and P. W. Morgan. (XIII, p. 129.)

Multiple Culture Technique for testing Rot Resistance in Jute. S. N. Basu, A. P. Bhattacharyya, and R. G. Bose. (XIV, p. 132.)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

PATENTS

Treating Cellulosic Raw Materials. J. C. Seailles. *B.P.* 647,129.

Raw cellulosic products are treated, as in *B.P.* 638,496, with hot water under pressure in an autoclave at 160–210°c., the time of treatment (90–270 min. at 160°c.) being adjusted according to the nature of the material treated and the ratio between the quantity of water and the quantity of material treated, the time being reduced to one half or less for each rise in temp. of 10°c. There is enough water to immerse, sprinkle, or wash the raw material in order to dissolve the soluble products resulting from the treatment, yielding a cellulosic pulp and a sugar solution rich in organic matter. Spent liquors from a previous treatment or wash water from the cellulosic products may be used partly or wholly to ensure shorter treatment and more concentrated organic solutions. The acidity of the liquid may be partly neutralised before or during treatment; e.g. strong acid may be added to bring the acidity of the initial liquid to $pH > 2{\cdot}5$ and under 0·1 N., or weak acid added to reach 0·2 N. to shorten treating time, e.g. acetic acid to $pH > 4{\cdot}7$. A pH-stabilising salt, e.g. sodium or calcium phosphate, may be incorporated, and the acidity may be controlled by injecting neutralising salts. The raw material may be pretreated with hot water, and the recovered solid products may be treated with milk of lime to facilitate and cheapen any bleaching and refining operations. S. V. S.

Cellulosic Materials of Improved Resistance to Chemicals, especially Alkalis. U.S. Secretary of Agriculture and R. E. Reeves. *U.S.P.* 2,520,963.

When cellulosic materials are treated with an anhydrous alcohol containing an anhydrous acid or an acid containing very little water as catalyst, some bonds in the cellulose molecule are broken and a modified cellulose is obtained containing chemically combined alcohol. The product shows remarkable resistance to cold or hot dilute aqueous alkalis. C. O. C.

Coating of Paper. H. P. Andrews Paper Co. *B.P.* 647,197.

Paper made substantially waterproof by sizing is prepared for the reception of an ink, dye, or sensitising solution by applying a colloidal aqueous dispersion of silica to give a surface coating of discrete submicroscopic particles of silica, yielding enhanced contrast between the paper base and the inked or dyed areas, e.g. in blue-printing. The silica coating may be formed by applying a silicate coating and then treating with alum to precipitate fine particles of silica on the paper. S. V. S.

Coated Paper. S. D. Warren Co. and F. H. Frost. *U.S.P.* 2,520,900.

The tendency for uneven and undue absorption of the top coating into the undercoat in twice-coated papers, particularly where the mineral component of the top coating comprises finely divided metal, e.g. Al, can be controlled by incorporating a finely dispersed waxy material of low acid value into the undercoat. The amount of the waxy material, e.g. paraffin wax, should be enough to ensure uniform hold-up of the top coating but not so much as to render the undercoat difficultly wettable by the top coating. C. O. C.

Treating the Surfaces of Porous Webs. Institute of Paper Chemistry, G. D. Knight, and B. W. Rowland. *U.S.P.* 2,521,666.

The web, e.g. paper, is passed on an impervious backing to a zone where the fluid ingredients are evacuated from and within the surface of the web, after which the coating or other liquid is applied to the evacuated portion of the web under at least atmospheric pressure and before direct exposure of the evacuated region to the ambient atmosphere. C. O. C.

Record Material or Manifold Sheet. National Cash Register Co. *B.P.* 646,926.

A record material insensitive to atmospheric conditions consists of a base sheet coated with a solid, e.g. gelatin, methyl cellulose, or casein, in which is dispersed a marking fluid consisting of a colouring matter and a low-viscosity, non-hygroscopic, water-immiscible liquid. On laying the coated side of the sheet on an underlying sheet and applying pressure, e.g. with a pencil, the solid coating is ruptured and releases the dispersed drops of marking fluid, so that they contact the underlying sheet C. O. C.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

PATENTS

Desalting Pickled Hides and Skins. C. G. Schneider and Hall Laboratories Inc. *U.S.P.* 2,516,806.

Inordinate swelling and gelatinization is avoided by drumming the skins in 2–6 times their weight of water containing 0·5–1·0% on their weight of an alkali-metal phosphate having an alkali-metal oxide : P_2O_5 ratio of 0·4–1·7 : 1, and crystalline ammonium phosphate (ratio of $(NH_4)_2O : P_2O_5$ of 1 : 1). C. O. C.

Proofing of Furs against Insects. Cyril Eastman (Furs) Ltd. and C. Eastman. *B.P.* 647,069.

A finely divided material is wetted with a solution or dispersion of an insecticide or suitable proofing agent, after which the furs are drummed with this material. Preferably sawdust wetted with a petroleum-spirit solution of D.D.T. is used. C. O. C.

XIII—RUBBER; RESINS; PLASTICS

Polyamides from Azelaonitrile and Formaldehyde. D. T. Mowry and E. L. Ringwald. *J. Amer. Chem. Soc.*, **72**, 4439–4441 (Oct. 1950).

Polyamides are formed by reaction of dinitriles with formaldehyde in presence of an acid catalyst. Details are given of the effect of various reaction variables on the mol. wt. of polymethyleneazelamide obtained from azelaonitrile. C. O. C.

PATENTS

Casein Plastics. B.X. Plastics Ltd., V. I. Furness, P. G. T. Hand, and A. E. Judd. *B.P.* 645,461.

Casein plastics of lower sensitivity and higher resistance to moisture are made from a casein–metal complex

obtained by treating a solution of raw casein in a water-soluble base, e.g. NaOH, with a salt of zinc, cadmium, or aluminium. W. G. C.

Emulsion Polymerisation of Ethylene. L. Seed and I.C.I. Ltd. *B.P.* 645,088.

Polymers and copolymers of ethylene with good mechanical properties are prepared in the form of stable emulsions by polymerising at 120–250°c. and 800–2000 lb./sq. in. in presence of water and an organic sulphonic acid or derivative, e.g. sodium oleo-*p*-anisididesulphonate, and a water-soluble salt of a polymer (mol. wt. > 300) of a carboxylic acid, e.g. ammonium polymethacrylate or sodium cellulose glycollate. The products are suitable for treating textiles. E. C.

Plasticisers for Vinylidene Chloride Copolymers. R. A. Scheiderbauer and du Pont. *U.S.P.* 2,518,442.

Polyoxyalkylene ethers of hexitol ring dehydration products, e.g. a sorbitan mono-oleate polyoxyethylene derivative, are good plasticisers for vinylidene chloride copolymers. C. O. C.

Carbon Black as Catalyst for Curing Alkyd Resin Adhesive Compositions. Johnson & Johnson (Gt. Britain) Ltd. *B.P.* 645,197.

Mixtures of an alkyd resin with a smaller quantity of an alcohol-modified urea or melamine resin are rapidly cured on heating when 0·2–5·0% of an alkaline (pH 7–10) colloidal (particle size 25–100 mμ.) carbon black is employed as catalyst. Such compositions do not cure in the cold, and are suitable for use in bonding unwoven webs and in adhesive tapes. E. C.

Aryl Phosphate Resins. H. Zenftman, A. McLean, and I.C.I. Ltd. *B.P.* 644,468.

Clear, heat-resistant and non-inflammable resins suitable for use in surface coatings and cements are prepared by heating an aryloxyphosphoryl dichloride with an equimolar amount of an aromatic compound having two phenolic hydroxyl groups not in the *ortho* position. E. C.

Synthetic Linear Polyamides containing Intralinear Cyclic Groups. D. C. Pease and du Pont. *U.S.P.* 2,516,585.

The copolymers of terephthalic acid or an amide-forming derivative thereof and bis-4-amino-3-methylcyclohexylmethane have high clarity, transparency, and stiffness. They are insensitive to water, and have a high softening point. They are useful in making bristles and as coatings. C. O. C.

Polyamide Polymers. W. A. Jordan, S. H. Shapiro, J. M. Schrager, and Armour & Co. *U.S.P.* 2,518,148.

Hard products of high m.p. and excellent protective-coating properties are obtained by treating a high-mol.wt. polyene fatty acid substance, e.g. linoleic acid, with a low-mol.wt. aliphatic diamine, e.g. ethylenediamine, to form a diamide, which is then heat-polymerised. C. O. C.

Colouring Aqueous Emulsions of Vinylidene Polymers. Monsanto Chemical Co., H. F. Park, and F. J. Pokigo. *U.S.P.* 2,520,429.

An amine is added to a solution of an organic dye in an aliphatic acid of 6–20 C, after which the solution is emulsified in water. This emulsion may be added to an aqueous emulsion of a vinylidene polymer without causing any tendency for premature coagulation. The dye is thoroughly dispersed throughout the resin, and in resolving the emulsion and drying the polymer the latter is not unduly soft and does not tend to shrink when stored for a long time. C. O. C.

Cross-linked Polymeric Materials. du Pont and P. W. Morgan. *U.S.P.* 2,520,609.

Derivatives of dithiocarbamic acid in which the substituents contain < 9 C are used as cross-linking agents for polymers containing a functional group; e.g. hydroxyethyl cellulose acetate–chloroacetate is insolubilised by cross-linking it by treating at 100°c. with thiuram sulphide. C. O. C.

Tetrachlorophthalic Acid and Anhydride— Curing of Aminoplasts. American Cyanamid Co. and E. F. Morfit. *U.S.P.* 2,520,100.

A mixture of tetrachlorophthalic anhydride and an inhibitor, e.g. hexamethylenetetramine, added to a standard urea- or melamine–formaldehyde moulding composition, confers excellent storage characteristics combined with rapid curing upon heating. C. O. C.

Aliphatic Cyano–Azo Compounds— Polymerisation Catalysts. du Pont and J. E. Castle. *U.S.P.* 2,515,628.

Aliphatic cyano-azo compounds of formula—

$$R^1{\cdot}CHR^2{\cdot}CH_2{\cdot}CR^3(CN){\cdot}N{:}N{\cdot}CR^3(CN){\cdot}CH_2{\cdot}CHR^2{\cdot}R^1$$

(R^1, R^2, R^3 = alkyl, preferably of < 4 C) are very effective catalysts in polymerisation reactions involving compounds containing ethylenic double bonds. Thus $\alpha\alpha'$-azobis(α-*iso*butyl-γ-methylvaleronitrile) is prepared by the reactions—

$$2\,Bu^t{\cdot}CO{\cdot}Bu^t + NH_2{\cdot}NH_2$$

$$\downarrow$$

$$Bu^t{}_2C{:}N{\cdot}N{:}CBu^t{}_2$$

$$\downarrow \text{HCN}$$

$$CN{\cdot}CBu^t{}_2{\cdot}NH{\cdot}NH{\cdot}CBu^t{}_2{\cdot}CN$$

$$\downarrow Cl_2$$

$$CN{\cdot}CBu^t{}_2{\cdot}N{:}N{\cdot}CBu^t{}_2{\cdot}CN$$

(Bu^t = $-CH_2{\cdot}CH(CH_3)_2$). It acts as a catalyst in the polymerisation of e.g. acrylonitrile. E. S.

Polysiloxane Resins. British Thomson-Houston Co. Ltd. *B.P.* 647,322.

Resinous polysiloxanes are obtained by treating a mixture of hydrocarbon-substituted halogenosilanes, of hydrocarbon : Si ratio 1·1·5 and containing a mono-hydrocarbon-substituted trihalogenosilane, dissolved in dioxan and/or trioxan and/or lower dialkyl ethers of ethylene or diethylene glycol, with a tertiary saturated aliphatic alcohol of 4–6 C. They may be used for coating or impregnating textiles or paper, or mixed with fillers as moulding compositions. C. O. C.

Acceleration Vulcanising Agents suitable for use in presence of Furnace Blacks. Monsanto Chemicals Ltd. and W. M. Morgan. (III, p. 121.)

Composition of Crotyl Cellulose and Compatible Butadiene Copolymer. du Pont and W. H. Charch. (VI, p. 126.)

XIV—ANALYSIS; TESTING; APPARATUS

Standardisation of Testing Methods for Textile Auxiliary Products. G. Schwen. *Melliand Textilber.*, 31, 627–630 (Sept. 1950).

This is a report of the work of the technical committee of the Arbeitsgemeinschaft Textilhilfsmittel-, Lederhilfsmittel-, und Gerbstoffindustrie. Work on the evaluation of wetting agents in an attempt to obtain a satisfactory testing method is described, and the work is being extended to desizing preparations, and water-repellent and anti-swelling agents. It is hoped ultimately to classify all these auxiliaries on the basis of their composition, properties, and application. H. K.

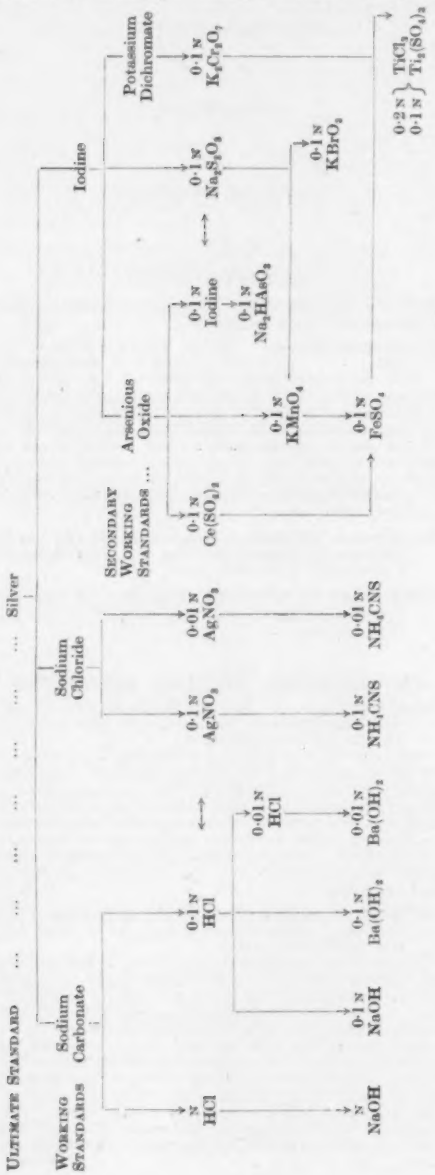
Method of Measuring Penetration and Wetting. C. R. Trommer. *Amer. Dyestuff Rep.*, 39, P 811–P 812, P 830 (27th Nov. 1950).

A method for measuring the penetrating as distinct from the wetting power of a surface-active agent consists in placing a steel block ($2\frac{1}{2}\times2\frac{1}{2}\times2\frac{1}{2}$ in.), with a $\frac{1}{4}$ in. diameter hole drilled vertically through it, on top of a number of layers of fabric (3 × 3 in.). The test solution (10 ml.) is added through the hole, and the time to penetrate the pad, judged by the closing of an electric circuit, is measured. To eliminate the effect of lateral wetting of the top layer, the time required to penetrate a single layer is determined separately and deducted from the original value. This corrected value is divided by the number of layers below the top in the test pad to give the time of penetration per layer. W. K. R.

The Standardisation of Volumetric Solutions. I.C.I. Ltd. Analytical Chemists' Committee. *Analyst*, **75**, 577–604 (Nov. 1950).

Full details are given of the methods which have been used by I.C.I. for a number of years for the preparation and standardisation of volumetric solutions. The titre of all standard solutions is based on the ultimate standard, pure electrolytic silver, through working standards—sodium carbonate, sodium chloride, and iodine— which are referred directly to the ultimate standard. In cases where the solutions are not directly referable to the working standards, secondary working standards— arsenious oxide and potassium dichromate— are employed. The standardisation scheme is given in previous column.

W. K. R.



Resistance of Wool Oils to Oxidation in Storage. A.A.T.C.C. Tentative Test. *Amer. Dyestuff Rep.*, **39**, P 911 (25th Dec. 1950).

An accelerated wool-oil oxidation test is described. Each test specimen (10 g.) is saturated with 10% of its weight of one of the oils to be tested, by immersing in an ethereal or ether–alcoholic solution of sufficient bulk to permit complete and even adsorption by the wool and allowing excess solvent to evaporate. The specimen is heated for 3 weeks at 120 ± 2°F. in a 1-pint closed glass jar, after which one half and a freshly oiled sample are washed in the Launder-Ometer, dried, and compared for degree of discoloration.

J. W. B.

Cryoscopic Estimation of *o*- and *p*-Cresol in Industrial Cresol. G. Vivier. *Chim. et Ind.*, **64**, 699–703 (Dec. 1950).

Commercial cresols contain variable amounts of three isomers, *o*-, *m*-, and *p*-cresol, together with certain impurities. With a fairly pure product it is possible to estimate the *o*- and *p*-cresol by cryoscopic methods. A study of variations in the crystallising point as a function of the variation in the amounts of the three isomers present has resulted in an equation which permits the percentages of each isomer to be calculated.

B. K.

Dienometric Estimation of Anthracene. (The late) A. F. Titley, J. F. Grove, and A. J. Allmand. *J.S.C.I.*, **69**, S 23–S 27 (Supplementary Issue No. 1, 1950).

A method is described for the estimation of anthracene involving the Diels–Alder condensation of the hydrocarbon, after removing tar acids and bases, with maleic anhydride by refluxing in xylene solution, followed by removal of the solvent by steam distillation and back titration of the unreacted maleic acid. The elimination of sources of error is outlined, and the application of the method to the determination of anthracene in technical pastes and oils is discussed.

W. K. R.

Determination of *p*-Nitrophenol. R. M. Lollar. *J. Amer. Leather Chem. Assocn.*, **45**, 728–733 (Nov. 1950).

A method for the determination of *p*-nitrophenol in leather is reported which depends on the development of an indophenol blue colour. The intensity of colour is preferably determined by means of a spectrophotometer, the accuracy of the method then being such that the degree of extraction from the leather is the limiting factor. The method is applicable to all leathers without isolating the *p*-nitrophenol from other components of the extract.

A. S. F.

Bibliography on Carotene Estimation, with special reference to Green-leaf Material. N. T. Gridgeman. *Analyst*, **75**, 574–576 (Nov. 1950).

Density Gradient Tubes in Fibre Studies and Analysis of Fibre Mixtures. J. M. Preston and M. V. Nimkar. *J. Textile Inst.*, **41**, T 446–T 454 (Nov. 1950).

A simple method is described for the quantitative analysis of binary mixtures of fibres in a density gradient tube, made conveniently from e.g. a 500-ml. stoppered measuring cylinder, and calibrated by having in suspension a number of glass floats, in turn calibrated beforehand. A dehydrating head, including devices for release of the specimen, may be fitted to the tube. Bad visibility due to fibres having a refractive index close to that of the liquid is overcome by using crossed Polaroid screens. Suitable liquid mixtures are e.g. xylene or heptane (light) with carbon tetrachloride or ethylene dibromide (heavy), while solid heavy components may be used provided they are readily soluble in the light component, e.g. chloral hydrate in water. The tube is filled by starting off with one component, adding portions of progressively varied composition, and standing 2 days to attain uniform gradient. A binary fibre mixture is dropped in, its level is observed, and from the known densities of the component fibres the percentage composition may be determined from a simple formula. It is considered that this method may be of particular value not only for mixtures such as viscose–casein, cotton–asbestos, nylon–wool, and rabbit fur–wool, but also in determining the amount of tin weighting on silk, or in controlling aqueous delustring of cellulose acetate, both of which processes alter the density.

J. W. B.

Simplified Method for Determination of the Oil Content of Textiles. A.A.T.C.C. Northern New England Section. *Amer. Dyestuff Rep.*, **40**, P 30–P 32 (8th Jan. 1951).

The standard method of determining oil and fat content of textiles is time-consuming and involves expensive and fragile apparatus. A new, simple, and equally accurate method, requiring 30 min., is described. A sample (10 g.) of material is weighed, placed in a 250-ml. beaker, and worked with 50 ml. of CCl_4 for 3–4 min., using the base of a 50-ml. cylinder as a plunger. The sample is pressed, and slightly > 25 ml. of expressed solvent is run into a 50-ml. beaker, measured in a 25-ml. cylinder, and transferred to a tared 100-ml. beaker. After gently evaporating the CCl_4, the beaker is reweighed. Twice the weight of residue gives the amount of oil in the sample. J. W. B.

Spot Test Analysis of the Ash of Natural Cellulosic Fibres. N. A. Matlin. *Amer. Dyestuff Rep.*, **40**, 44–48 (22nd Jan. 1951).

A system of spot tests applied to the ash of native cellulose fibres is developed and described together with discussion on the chemical aspects. Included in the tests, not all of which are found satisfactory, are borates, chromates, phosphates, sulphates, silicates, Fe, Sb, Sn, Cu, Zn, Al, Mg, Ca, and Si. J. W. B.

Role of the Cationic Ash of Jute Fibre in its Acid Value Determination. H. Chatterjee. *J. Textile Inst.*, **41**, T 454 (Nov. 1950).

Correction to previous paper, *J. Textile Inst.*, **41**, T 243–T 248 (July 1950): J.S.D.C., **66**, 665 (Dec. 1950).

Photoelectric Instrument for Measuring Tippiness in Wool Staples. H. Goot and R. E. Belin. *J. Textile Inst.*, **41**, T 455–T 465 (Dec. 1950).

Tippiness is due to variations in fibre length, and may be estimated by measuring these photometrically. The wool is teazed out, clamped at one end, laid lengthwise on an optically black tray which can move parallel to the staple length, and covered with a thin sheet of clear glass. An evenly illuminated light image 3 in. × ¼ in. is projected on the wool at right-angles to the direction of movement, and the amount of scattered light is measured by a photocell. Scatter depends on the number of illuminated fibres, the nature of their surface, and their cross-section. Sampling methods, interpretation and presentation of results, and possible improvements in the device are discussed. J. W. B.

Cordage Testing Methods. IV— Proof Testing of Rope. D. Himmelfarb. *Fibres*, **17**, 398–400 (Dec. 1950).

American and British methods for evaluating the flexibility (stiffness), twist, buoyancy, and waterproofing of ropes are discussed. C. O. C.

Proposed Tentative Test Methods for Evaluation of Continuous Scouring of Raw Grease Wool. A.A.T.C.C. Wool Scouring Committee. *Amer. Dyestuff Rep.*, **39**, P 813–P 815 (27th Nov. 1950).

A standard apparatus and set of operating conditions are specified for purposes of calibration; otherwise each may be varied to suit a particular purpose. For calibration, twenty 5-g. hanks of a standard grease wool yarn are successively scoured by immersion and agitation for 30 sec. in (1) a suint bowl containing 2 litres distilled water, (2) a scour bowl containing 3 g. soap flakes and 25 g. soda ash dissolved in 2 litres distilled water, and (3) running distilled water, all at 140 ± 2°F. After each bowl, the hanks are squeezed through a wringer, the squeeze liquor from bowl (2) being returned. The hanks are then dried for 4 hr. at 140°F., and rapidly extracted with cold carbon tetrachloride by working in a brass cylinder and piston system. An aliquot of the solvent is evaporated in a tared aluminium dish, which is then weighed, and the residual grease content of each hank is calculated. Residual grease content is plotted against the cumulative weight of wool scoured. W. K. R.

Foveal Hue Discrimination in the presence of a White Surround. R. A. Weale. *Nature*, **166**, 872–873 (18th Nov. 1950).

The hue discrimination in the fovea at an intensity of 0·95 e.f.c. has been measured in the case where the test field (subtending an angle of 50′) was surrounded by a white field. The results are presented in graphical form with the least perceptible difference $\Delta\lambda$ plotted against λ. There are a number of optima and pessima for which there is no full explanation on existing hypotheses. These are discussed, and it is concluded that the explanation must be found on the basis of a nervous mechanism possibly due to the interaction of the receptor paths. W. J. M.

Investigations on the Passage of Light through Suspensions. A. H. M. Andreasen, K. Krebs, N. A. Dalsgaard-Pedersen, E. Camradt-Petersen, and B. Kjær. *J.S.C.I.*, **69**, 312–317 (Oct. 1950).

The light transmission and scattering in aqueous suspensions have been examined. Monodisperse suspensions of barium sulphate were prepared in the range 0·1–4·0 μ. The transmission and scattering showed a very pronounced maximum at particle sizes of 0·6–0·8 μ. Three light filters were used— red, green, and blue— the greatest effect being obtained with the blue. The absorption maximum is also moved to smaller particle sizes at shorter wavelength. Monodisperse fractions from ground barytes, chromic oxide, and pyrolusite were obtained by elutriation. Similar results were obtained, but with the coloured particles the absorption maxima occur at smaller particle size. W. J. M.

Fluorescent Lamp Artificial Daylight Units for Colour Matching. G. T. Winch, W. Harrison, and H. R. Ruff. *Trans. Illuminating Eng. Soc.*, **16**, 1–12 (Jan. 1951).

The good colour and colour-rendering properties of the fluorescent lamp, its high efficiency, and the convenience of its form for obtaining high even illumination over large areas make it attractive for viewing and comparing large colour samples in spite of the small departures from the ideal black-body spectral distribution. A colour of approx. 6500°K. has been preferred for fluorescent lamps for colour matching. Cold-cathode lamps have been in industrial use for some years for critical colour-matching purposes, while hot-cathode colour-matching fluorescent lamps are now commercially available. In addition a unit combining blue-fluorescent and tungsten-filament lamps has been devised. This provides slightly closer approximation to black-body spectral distribution provided care is taken to ensure adequate mixing of the light, and adjustment made for the differential life behaviour of the component lamps. C. O. C.

Measurement, Representation, and Specification of Colour and Colour-rendering Properties of Light Sources. G. T. Winch and H. R. Ruff. *Trans. Illuminating Eng. Soc.*, **16**, 13–25 (Jan. 1951).

Statement of the problem is followed by a description of methods of measuring and specifying colour and colour-rendering properties with particular reference to tubular fluorescent lamps. It has been found desirable in Great Britain to specify the colour characteristics of these light sources in terms of both their colour-rendering properties expressed as percentage luminance in eight spectral bands and their colour appearance in C.I.E. chromaticity co-ordinate limits. Typical forms of colour and spectral band specification are indicated, and a preferred form of block diagram is given in which the percentage luminance values in the eight spectral bands are plotted on logarithmic ordinates. New and more accurate measuring apparatus is described which enables lumens, trichromatic co-ordinates, and luminance in eight spectral bands to be measured directly on the C.I.E. system in quick succession in the one apparatus. The accuracy of such precision instruments may make it possible ultimately to specify colour-rendering properties in terms of percentage luminance in spectral bands alone. C. O. C.

Work of the German Fastness Commission. W. Weltzien. *Melliand Textilber.*, **31**, 634–636 (Sept. 1950).

In February 1949 the German Fastness Commission (D.E.K.) was reconstituted as a sub-committee of the Working Committee C 9 in the Fachnormenausschuss für Materialprüfung. The fastness tests discussed were fastness to washing, sea water, and perspiration, and also methods of assessing the results. A meeting of representatives of the Swiss, French, and German Fastness Commissions was held in February 1950 in Basle to consider further testing methods. H. K.

Observations on I. H. Godlove's *Uniformity of Grading of the American, British, and German Light-fastness Standards.* Society of Dyers and Colourists. *Amer. Dyestuff Rep.*, **39**, P 912 (25th Dec. 1950).

In a criticism of Godlove's paper (*ibid.*, **39**, P 215 (3rd April 1950)), the Society's Fastness Tests Co-ordinating Committee expresses dissatisfaction with the merits of the quantities calculated by applying the Adams' "chromatic value" formula on the grounds that the Committee has no experience of the relation between the results obtained thereby and visual judgment of fading. American standards are, moreover, based on a different concept and the criterion of fading is also different. American standards give satisfactory grading when exposed for one time only, but this is not suitable for dyeings which show a small initial fade but which thereafter remain stable. Although the British ideal that each standard should require twice the time of exposure to fade required by the previous standard has not been quite attained, this has not invalidated the use of the scale in practice. Examples are given of inconsistencies in the paper under criticism.

J. W. B.

Test for Fastness to Perspiration. Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinie A 2524. *Textil-Rund.*, **6**, 25 (Jan. 1951).

A standard method for estimating fastness to perspiration is given in which the samples are treated first in an alkaline and then in an acid salt solution. F. A.

Tests for Fastness to Chlorine [Bleaching]. Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinie A 2557. *Textil-Rund.*, **6**, 27 (Jan. 1951).

Standard tests are described in which there are two degrees of treatment using 0·5 g. and 2·0 g. active chlorine respectively per litre. F. A.

Test for Fastness to Peroxide Bleaching. Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinie A 2554. *Textil-Rund.*, **6**, 26 (Jan. 1951).

A standard method for estimating fastness to peroxide bleaching is described. Different recipes are given for the peroxide liquors employed with cellulose fibres, wool, and silk. F. A.

Test for Fastness to Neutral, Acid, and Chrome Cross Dyeing. Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinie A 2576. *Textil-Rund.*, **6**, 28 (Jan. 1951).

Details of a standard test including recipes for the various blank dyebaths. F. A.

Multiple Culture Technique for testing Rot Resistance in Jute. S. N. Basu, A. P. Bhattacharyya, and R. G. Bose. *J. Textile Inst.*, **41**, T 466–T 480 (Dec. 1950).

A method is described for the evaluation of rot resistance in textiles, particularly jute materials. To avoid fungal specificity towards antiseptics, samples are inoculated simultaneously with 5 species of fungi which occur frequently in mildewed jute and which possess marked tendering powers. Conditions are evolved which favour rapid culture growth and loss in strength, being comparable with soil incubation. Fabric proofed with copper naphthenate is superior in rot resistance to cuprammonium-treated fabric containing more copper; this, as leachability is similar, supports previous views that the naphthenate radical itself has some toxicity. Cuprammonium treatment reduces the fabric strength, while sodium pentachlorophenate is both highly leachable and unsuitable at the concentration used. J. W. B.

Spray Test for the Evaluation of Waterproof Properties. Schweizerischer Verband für die Materialprüfungen der Technik, Kommission 25. Richtlinie D 1332. *Textil-Rund.*, **5**, 463–464 (Nov. 1950).

Full details are given of a standard test in which the water uptake of samples is measured after spraying with water for 1 min. F. A.

Determination of Injurious Copper in Textiles to be Rubberised. M. W. Weiss, E. Hoffmann, and S. Lee. *Amer. Dyestuff Rep.*, **40**, 41–43 (22nd Jan. 1951).

Although even minute amounts of "free" or "ionic" copper are injurious to rubber, the copper in certain textile colouring matters, e.g. copper phthalocyanine, is so firmly combined that it is quite harmless. Present testing methods involve complete destruction of the sample and inclusion of this harmless portion in the analysis, thus excluding e.g. a phthalocyanine-dyed fabric from use in rubber coating. To determine harmful copper only, a sample (5 g.) is extracted with N. nitric acid, and the extract is made ammoniacal, treated with sodium diethyl dithiocarbamate reagent, and diluted in a Nessler tube to 100 ml., where it is compared with a series of tubes prepared from standard copper sulphate solutions. J. W. B.

PATENTS

Colorimeter. W. J. Boyer. *B.P.* 646,796.

Incorporating a water jacket having windows and filter carriers in a colorimeter with a single photoelectric cell enables a high-intensity lamp to be used and thus gives an instrument of good sensitivity and stability.

W. G. C.

Phase-contrast Microscopy. Cooke Troughton & Simms Ltd., E. W. Taylor, and B. O. Payne. *B.P.* 647,191.

Methods of obtaining change in phase and of controlling amplitude are described.

B.P. 647,207.

The reflector is mounted on the optical axis of the microscope between the phase plate and the objective, thus preventing the field of view from being flooded with light reflected from the surface of the phase plate. This renders coating of the phase plate with an absorbent metallic film unnecessary, and so avoids loss of incident light before it reaches the specimen. C. O. C.

Dielectric Determination of Moisture Content. J. L. Shaw. *B.P.* 647,990.

Opposite sides of a narrow rectangular box-like condenser serve as electrodes and comprise metal plates with inner faces insulated, a standard weight of material being pressed into the box by a shaped lid and acting as the dielectric. The condenser is in circuit with instruments for measuring the capacitance and/or power factor, the regain being read directly from a calibrated dial. J. W. B.

Abrasion-testing Machine. R. L. Fish. *U.S.P.* 2,519,556.

A machine is described in which the fabric to be tested is held on a rack and a series of abradant whipping elements rotate so as intermittently to strike and lash the fabric. C. O. C.

Sampling Device for Pressurised Package Dyeing Machines. P. M. Cole and T. W. Stricklin. (VIII, p. 126.)

XV— MISCELLANEOUS

PATENT

β-Hydroxybutyraldehyde for Preserving the Colour of Fruits and Vegetables. Zenith Processing Corpn. and H. S. Hateshi. *U.S.P.* 2,519,465.

β-Hydroxybutyraldehyde maintains the fresh appearance of fresh fruits and vegetables, prevents discoloration, and in some cases intensifies the natural green colour of the leaves. It is best used in a 0·01–0·5% aqueous solution.

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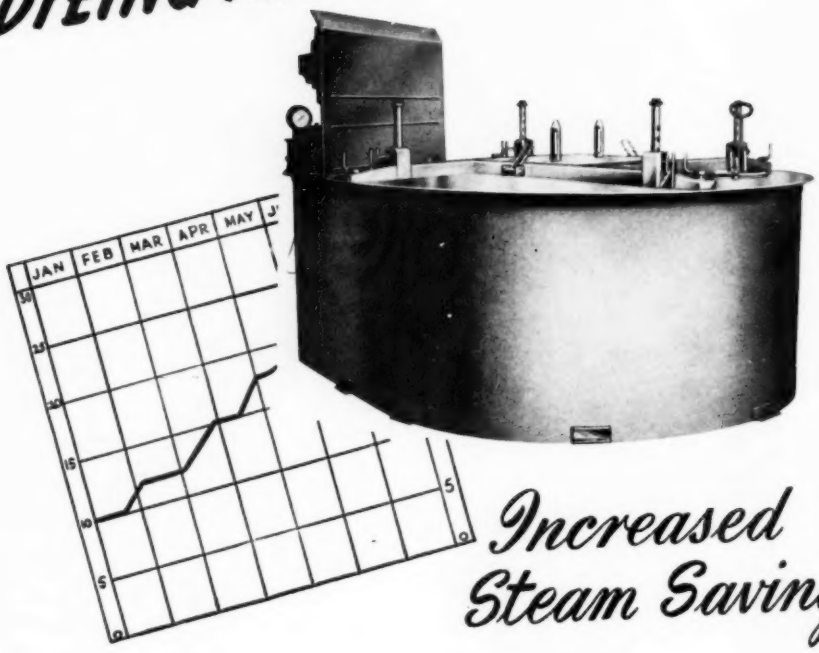

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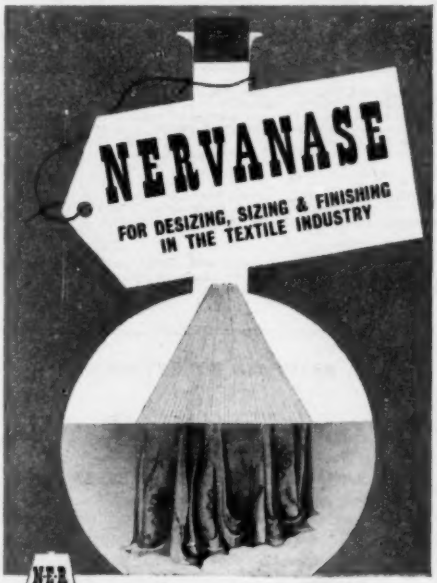

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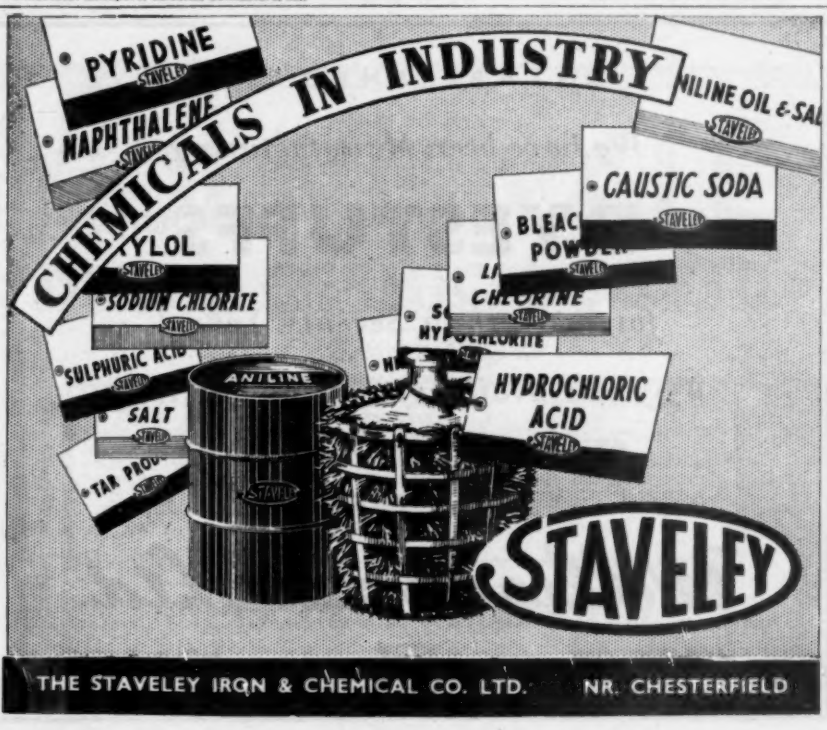

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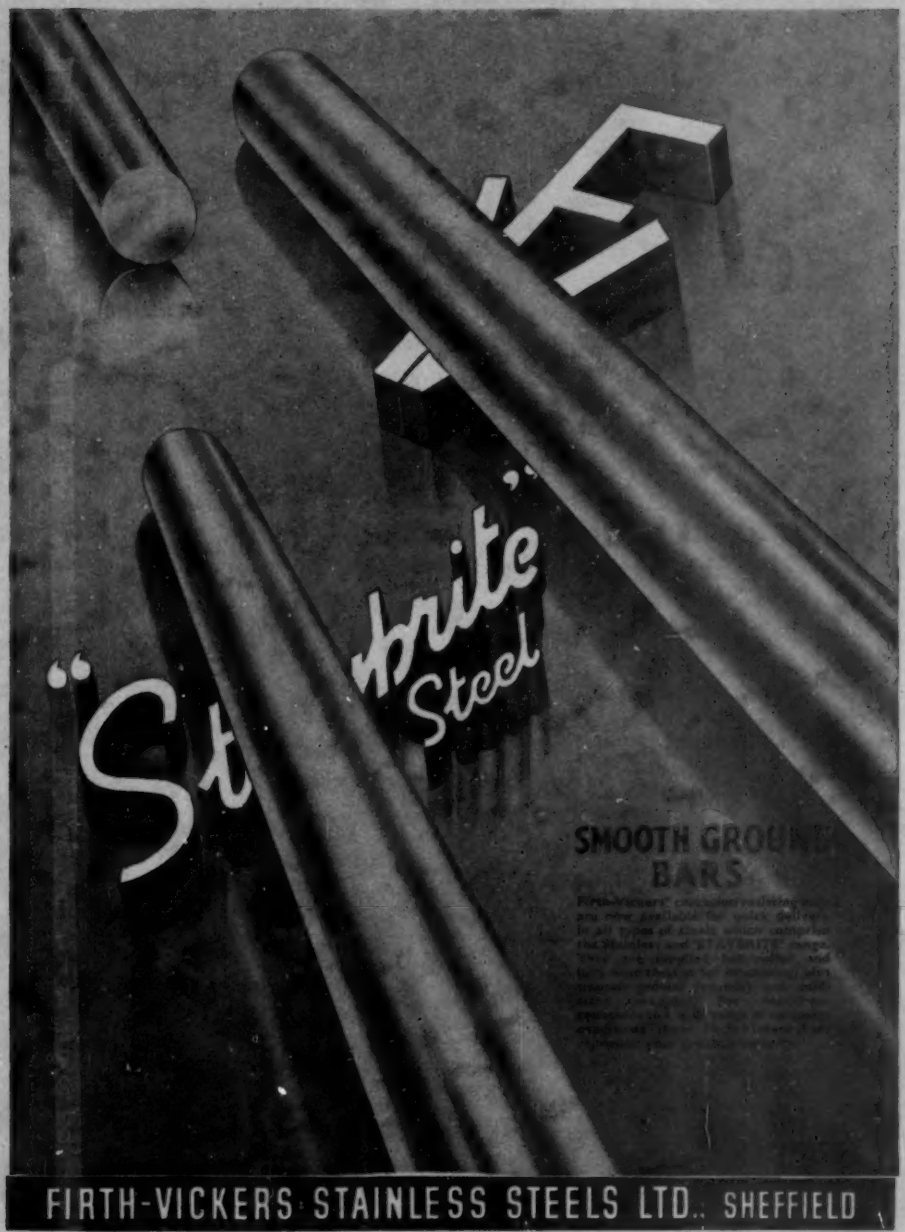
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